NITROGEN

No. 6 JUNE 1960

The Magazine of World Nitrogen

PUBLISHED BY

THE BRITISH SULPHUR CORPORATION LTD.
FISON HOUSE
95, WIGMORE STREET
LONDON, W.1.

Telephone : WELBECK 5500
Telegrams : SULFEX, TELEX, LONDON
Cables : SULFEX, LONDON

Chairman: Major-General G. E. WILDMAN-LUSHINGTON, C.B., C.B.E
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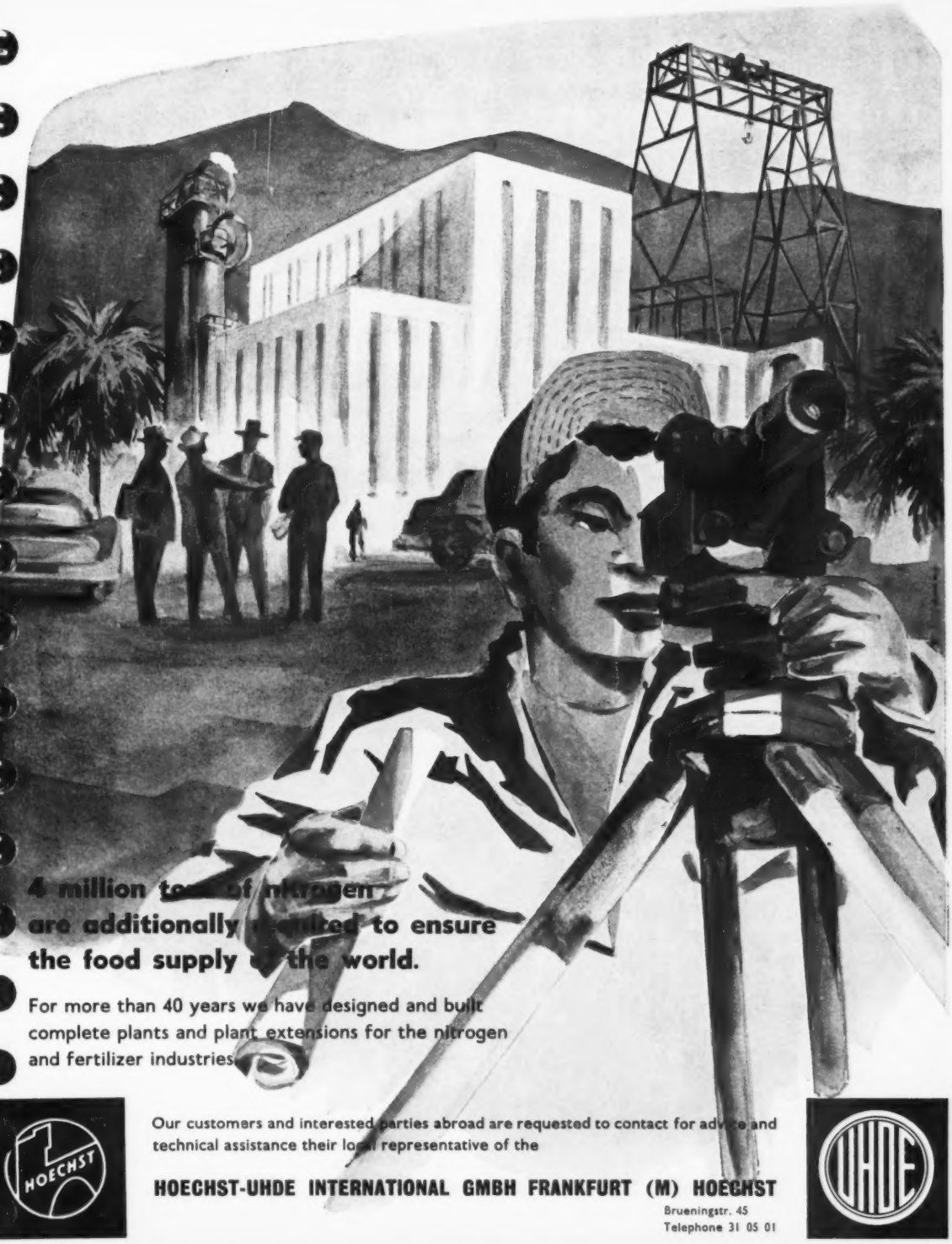

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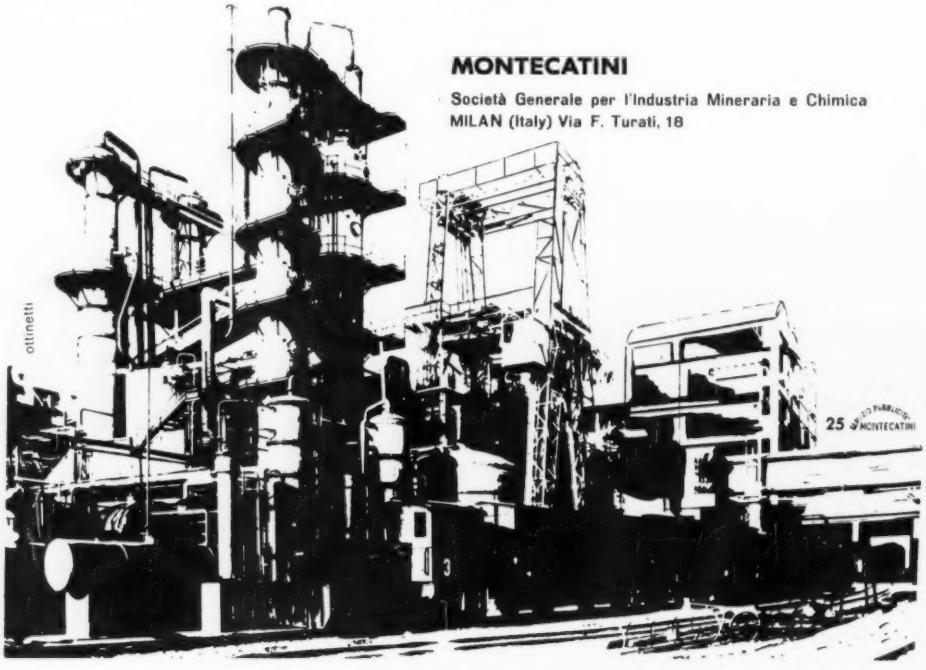

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Portugal's Nitrogen Industry

Present-day Aspects

COMPRISING some 34,500 square miles in area, Portugal has a population of nearly 9 million. Chief features of the country's economy are the agricultural industry which employs some 47% of the Portuguese working population and traditional industries based on the processing of natural products which group includes sardines, cork, box-board resin, glass and porcelain, and wine-making, and the textile industry which obtains its raw materials from the African overseas provinces. The modern industries are tyres, cement, electric cables and switch gear, petroleum refining, cellulose and paper. There is, too, some heavy industry based on steel which is due for considerable expansion.

Exports

As well as being an exporter of port wine, Portugal is one of the largest exporters of resin and turpentine; and important in respect of sulphur pyrites and wolfram. It is thus of importance to the chemical industry as a purveyor of raw materials. As is natural, however, with a raw materials exporting country, the course of Portuguese exports has been largely governed by two basic factors: limited industrial development, and concentration on a small number of products. These factors have been responsible for the increase in the instability of income from exports, while fluctuations of economic activity in industrial countries or the influence of political events on world commerce, e.g., the Suez crisis and the Korean war,

have always been reflected in the price and volume of buying of some of the principal Portuguese exports, particularly minerals. As an important part of the country's exports are raw materials as opposed to finished products the trend of expansion over a long period has not been so pronounced as in the case of industrial countries producing a greater variety of exports. Hindering Portugal's natural possibilities, too, has been the poor development of industry due to lack of coal and electric power.

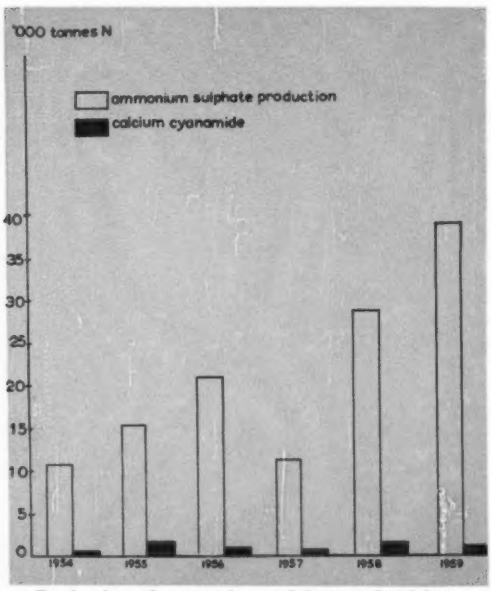
In September 1952 the Portuguese Government devised a six-year plan for development of the country and its overseas possessions which was to run from 1953 to 1958 and foresaw an investment in it of some 13,500 million escudos, more than half of which was to be spent on the colonies. The most important plans concerned agriculture, build-up of sea strength, erection of iron and steel works, development of transport and traffic, intensive prospecting for minerals and build-up of the

chemical industry. Because of its structure in Portugal, priority in the industrialisation programme has been given to the needs of agriculture, with the building-up of the fertilizer industry as one of the most important items of the Government's programme. (In 1954 Portugal was listed among the smallest chemical-producing countries in Europe.)

Nitrogen Industry

In July 1954 plans regarding the development of Portugal's nitrogen industry were revealed. Some £15 million was set aside for this purpose. To provide for the construction of a nitrate plant with an annual capacity of 80,000 tonnes a year and the expansion of ammonium sulphate production to 140,000 tonnes a year.

Production of nitrogen fertilizer in 1952/53 (fertilizer year) was 8,200 tonnes N consisting exclusively of ammonium sulphate. By the following year production had increased 50% and in addition to 11,700 tonnes N ammonium sulphate, some 2,000 tonnes N calcium cyanamide were produced. By 1954/55 Portuguese production of nitrogen amounted to 12,000 tonnes N as ammonium sulphate and consumption totalled 57,600 tonnes N, including besides ammonium sulphate other nitrogen fertilizers such as ammonium nitrates, calcium nitrate, sodium nitrate, etc. (See Table 2.) Since 1954 production of ammonium sulphate has steadily increased with the exception of the year 1957, and towards the end of the first Six-Year Plan increasing output of ammonium sulphate is noticeable (see Figure below). Consumption of nitrogen fertilizers was high in 1954 (57,600 tonnes), but rose further to reach a total of 65,100 tonnes in 1959.



Production of ammonium sulphate and calcium cyanamide in Portugal

In the manufacture of ammonia the use of hydroelectricity has been prominent and it is only in the Second Six-Year Plan, 1959 to 1965, that other raw materials, notably fuel oil, are achieving prominence.

Portugal's consumption of nitrogen products has shown a steady increase since 1954. Particularly marked has been the increased consumption of ammonium sulphate. (See Table 2.) Use of ammonium nitrates has fluctuated. In 1958 consumption rose 14,500 tonnes but fell to 9,600 in 1959. Sodium nitrate consumption has shown some decline also. Calcium cyanamide consumption, however, has remained fairly steady over recent years.

Development Plans

The First Six-Year Plan, which was completed in 1958, aimed at co-ordinating the development of Portuguese economy and speeding up certain lagging sectors. In the nitrogen industry these aims were:—

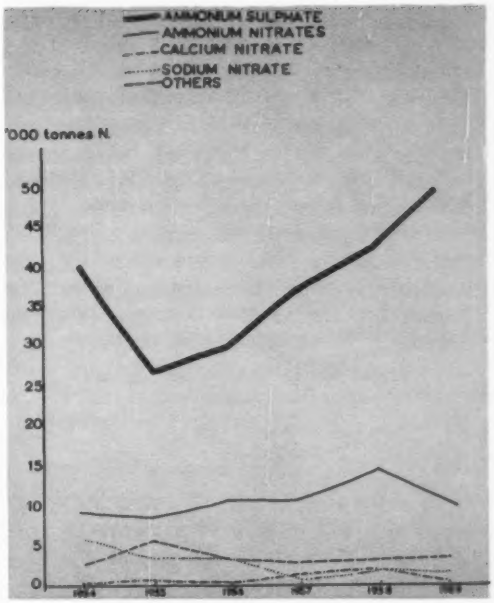
(a) Expansion of the works in Estarreja of Cia Ammoniaco Portugues, in order to raise the annual production of ammonium sulphate to 70,000 tonnes.

(b) Better utilisation of the ammonia works at Alfererradede and expansion of sulphate production at Barreiro, to provide a more or less equal production volume.

(c) Erection of a new plant with an annual production of 36,000 tonnes ammonia, which would serve for the manufacture of 80,000 tonnes of ammonium nitrate, and for fulfilment of Uniao Fabril do Azoto's production estimate for ammonium sulphate.

In effect, Ammoniaco Portugues surpassed in 1958 the target figure of 70,000 tonnes of ammonium sulphate a year, the ammonia works at Alfererradede have been expanded and Nitratos de Portugal provided the ammonia required for production of the 80,000 tonnes of ammonium nitrate.

The Second Development Plan embraces a further six years—1959-1964. Its objectives and the volume of expenditure envisaged will far surpass all that foreseen for the first Plan. The policy of the new Plan is the same, however, and is conceived as an instrument of State intervention in the national economy allowing full collaboration and combined effort of the Government and the nation so that expectations in regard to economic development in the Plan are fulfilled. Main sections in the Plan are: agriculture, industry, electricity, transport and communications, research and technical education. During the first year of this Second Six-Year Plan investments in Portugal and the overseas provinces were estimated at up to £66 million while all the undertakings provided for in the Plan will require, during its currency in the Metropolitan area and overseas, a total investment of 30,000 million escudos (£375 million). When considered in relation to the overall budget expenditure for 1960 of 9,500 million escudos (£118.75 million), this is indeed a high figure.



Fertilizer consumption in Portugal

A substantial expansion in the nitrogen industry is expected to meet an increasing part of the growing domestic demand.

Uniao Fabril do Azoto

Uniao Fabril do Azoto of Lisbon is a wholly-owned subsidiary of Companhia Uniao Fabril, who are engaged in the manufacture

Table I

PRODUCTION OF NITROGENOUS FERTILIZERS

(000 tonnes N)

	1954	*1955*	*1956*	*1957*	*1958*	*1959*
Ammonium sulphate	12.0	16.8	22.0	12.9	28.1	37.9
Calcium cyanamide	.4	2.6	1.9	1.1	2.2	2.1
Total	**12.4**	**19.4**	**23.9**	**14.0**	**30.3**	**40.0**

Table II

CONSUMPTION OF NITROGENOUS FERTILIZERS

Ammonium sulphate	40.3	27.5	29.9	37.1	43.8	49.9*
Ammonium nitrates	8.5	7.4	10.6	10.3	14.5	9.6*
Calcium nitrate	.4	.5	.2	1.9	2.3	.5*
Sodium nitrate	6.0	3.6	3.5	.5	2.6	1.9*
Calcium cyanamide	1.7	4.1	2.1	1.5	2.3	2.3*
Complex fertilizers	.6	.6	.9	.9	.9	.9*
Other	.1	.1	.1	n.a.	n.a.	n.a.
Total	**57.6**	**43.8**	**47.3**	**52.2**	**66.4**	**65.1***

*—Provisional n.a.—Not available

of a great number of products ranging from fertilizers, metals, oils to jute products. Uniao Fabril do Azoto operate works at Alfererradede near the River Zeyere Power Stations. Production started in 1952 and capacity is at present 19,000 tonnes N a year, based on use of electrolytic hydrogen. The 1948 Imperial Chemical Industries Ltd.'s version of the Haber Bosch process is used for ammonia synthesis. Some 700 tonnes of ammonia are sold a year in anhydrous or solution form, the balance being used in the manufacture of ammonium sulphate at Alfererradede and at Barreiro.

Production of Ammonium Sulphate

('000 tonnes N)

1952	... ...	4.4	1956	... ...	12.3
1953	... ...	3.9	1957	... ...	6.8
1954	... ...	6.7	1958	... ...	13.2
1955	... ...	9.0	1959	... ...	n.a.

Expansion plans are in hand by this company, which will build a 40,000 tonnes a year N ammonia plant at the Barreiro Works, due on stream in 1962. In addition the Barreiro Works will receive 15,000 tonnes of anhydrous ammonia a year, purchased from the Sociedade Portuguesa de Petroquimica.

The company has also been licensed to manufacture up to 70,000 tonnes a year of a compound fertilizer (as yet unspecified).

Within the last few weeks, Uniao Fabril do Azoto have signed an agreement with Società Montecatini, Milan, for construction at Lavradio, near Barreiro, on the River Tagus, of a large plant for the production of urea, using the Fauser-Montecatini processes. The new plant will be able to operate both full liquid-phase recycling, using all the ammonia output for urea production, and on partial recycling which gives, besides urea, various nitrogen compounds. This new plant will be Montecatini's 34th for production of urea. It is being engineered by M. W. Kellogg (International) Co.

Ammoniaco Portugues S.A.R.L.

Production was started in 1951 by Ammoniaco Portugues S.A.R.L. at works at Esterreja near Lisbon, having a yearly capacity of 13,000 tonnes N based on the use of electrolytic hydrogen. In 1958 an additional 10,000 tonnes N of capacity was brought into operation based on the use of gases from a neighbouring oil refinery.

To date ammonium sulphate is the only fertilizer manufactured, and production is estimated as follows:—

Estimated Production of Ammonium Sulphate

('000 tonnes N)

1952	... ...	4.0	1956	... ...	9.7
1953	... ...	5.0	1957	... ...	6.1
1954	... ...	5.3	1958	... ...	14.9
1955	... ...	5.8	1959	.. ...	n.a.

Extensions are being carried out by M. W. Kellogg (International) Co. to the Esterreja works which will raise total N capacity to 33,000 tonnes N by 1961. Some of the hydrogen used in the additional ammonia production will be based on the use of fuel oil. Ammonium sulphate capacity will be raised to 36,000 tonnes N a year and, in addition, facilities for production up to 13,000 tonnes N of ammonium nitrate and 10,000 tonnes N of urea will be set up.

Of a total cost for this expansion of 240 million Escudos (£3 million) the Portuguese Government are investing 120 million Escudos (£1½ million).

Nitratos de Portugal

Nitratos de Portugal was established in 1957 as part of the Second Six-Year Development Plan to manufacture, distribute and sell nitrogen fertilizers. The company's works are situated 19 miles east of Lisbon, and use ammonia produced by Sociedade Portuguese de Petroquimica. The latter will use gases from the Lisbon oil refinery operated by SACOR as raw material for a 40,000 tonnes N a year ammonia plant. Principal shareholders in Nitratos de Portugal are Ammoniaco Portugues, S.A. de Produits et Engrais Chimiques, the Companhia Portuguesa de Fornos Electricos, SACOR and the National Federation of Wheat Producers. The principal end-product of the new works will be lime ammonium nitrate (20,000 tonnes N a year). In addition to nitric acid, other products will be calcium nitrate (2,500 tonnes N a year) and ammonium phosphate or nitrophosphate. The company has also been licensed to manufacture up to 25,000 tonnes a year of a compound fertilizer (as yet unspecified).

Most of the installations are being supplied by the West German concern, Friedrich Uhde G.m.b.H.

Companhia Portuguesa de Fornos Electricos

Portugal's only producer of calcium cyanamide at present is Companhia Portuguesa de Fornos Electricos who started production of this nitrogen fertilizer in 1954. Output is as follows:—

Production of Calcium Cyanamide

('000 tonnes N)

1954	.4	1957	1.1
1955	2.6	1958	2.2
1956	1.9	1959	2.1

With an additional 3,000 tonnes N in the form of calcium cyanamide, Portuguese nitrogen capacity will reach a level of 135,000 tonnes N a year. This should make it capable of meeting virtually all domestic requirements and may even provide a substanial surplus for export.

Fertilizer Imports

The main fertilizer imported by Portugal is ammonium sulphate, the greater part being supplied by West Germany (37,507 tonnes in 1959), followed by Belgium and Luxembourg (22,393 tonnes in 1959) and Italy (12,083 tonnes in 1959). Imports of ammonium nitrates showed a sudden rise last year to 10,603 tonnes compared with 3,771 tonnes for the previous year. Calcium nitrate imports have fallen again from the 15,125 tonnes in 1958 and also those of calcium cyanamide (6,107 tons in 1954 as against 986 in 1959).

Table III

NITROGEN FERTILIZER IMPORTS

(tonnes)

	1954	*1955*	*1956*	*1957*	*1958*	*1959*
Ammonium Sulphate*						
United Kingdom	10,831	—	—	5,914	—	—
West Germany	44,889	21,137	16,766	46,387	39,034	37,507
Austria	13,155	9,010	4,335	6,448	—	—
Belgium and Luxembourg	17,162	11,859	9,243	24,239	14,810	22,393
Italy	21,389	16,112	15,041	18,602	11,188	12,084
Netherlands	33,362	100	—	22,795	1,485	—
France	-	—	—	—	19,855	—
Total	**140,738**	**58,217**	**45,384**	**124,385**	**86,363**	**71,984**
Ammonium Nitrates						
United Kingdom	690	612	884	666	—	—
West Germany	403	580	400	1,152	402	603
Austria	391	—	—	—	—	—
Belgium and Luxembourg	695	568	920	869	658	6,550
Italy	—	51	988	496	2,360	—
France	—	—	—	50	—	—
Netherlands	—	—	—	—	351	3,453
Total	**2,179**	**1,810**	**3,192**	**3,233**	**3,771**	**10,603**
Calcium Nitrate						
West Germany	2,712	3,344	1,272	10,884	8,316	1,069
Italy	—	—	50	—	1,188	—
Norway	—	—	—	980	4,040	—
Netherlands	—	—	—	—	1,581	1,976
Belgium and Luxembourg	—	—	—	347	—	—
Others	—	—	2	—	—	—
Total	**2,712**	**3,344**	**1,325**	**12,211**	**15,125**	**3,045**
Sodium Nitrate						
Chile	35,560	23,464	22,525	—	16,286	12,500
West Germany	3,350	—	—	3,163	1,134	495
Netherlands	69	—	—	—	—	—
France	—	—	—	5	—	—
Total	**38,980**	**23,464**	**22,525**	**3,168**	**17,420**	**12,995**
Calcium Cyanamide						
Belgium and Luxembourg	742	4,707	1,285	1,978	493	986
Italy	2,208	2,708	—	—	—	—
Norway	3,158	—	—	—	—	—
Total	**6,107**	**7,415**	**1,285**	**1,978**	**493**	**986**

*—Includes ammonium sulphate nitrate

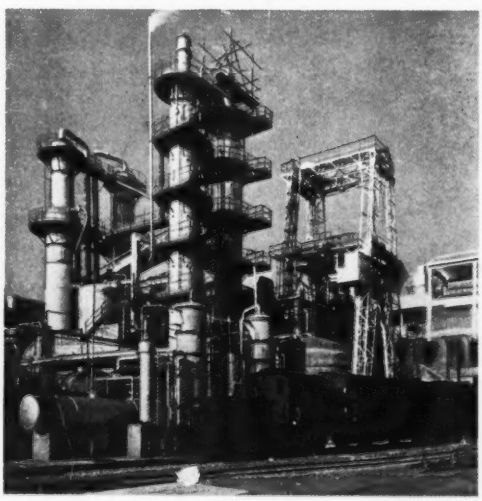
The large increase in Portuguese output of nitrogenous fertilizers, and ammonium sulphate in particular, is shown by the fall in imports of ammonium sulphate which in 1957 totalled 124,385 tonnes but dropped in 1958 to 86,363 tonnes and 71,984 tonnes in 1959. The United Kingdom and Austria, which in 1957 supplied 5,914 tonnes and 6,448 tonnes of sulphate respectively, lost their Portuguese markets entirely in 1958 and 1959. The Netherlands suffered the greatest loss, however, with Dutch exports falling from 22,795 tonnes in 1957 to 1,485 tonnes in 1958 and ceasing entirely last year. The increase in sodium nitrate imports since 1957 has resulted largely from a Portuguese-Chilean trade agreement in 1958.

Conclusion

The nitrogen industry in Portugal is still based primarily on the use of hydroelectric power, hence is closely related to price and availability of hydroelectric supplies. Examination of the average cost of electrical power used at the Alfererradede works of Uniao Fabril do Azoto show that these costs must be considered excessive for competition with similar imported fertilizer products. There has been some improvement in the situation, however, with the start of the Central Water Service at Picote in 1958.

To help domestic nitrogen producers in Portugal sell at prices competitive on the home market with imported fertilizers the Ministry of Economics has instituted subsidies. These subsidies, however, have been substantially reduced since 1956 and it is the level of prices which is considered to be the major stumbling block to increased fertilizer consumption by Portugal's agricultural industry. A particular point of contention with the Portuguese nitrogen producers such as Uniao Fabril do Azoto and Ammoniaco Portugues is that the Government has not allowed them any price concessions for power. So although Uniao Fabril do Azoto built their Alfererradede works between the power stations at Belver and Castelo do Bode, they receive no concessions in the price they pay for power, and Ammoniaco Portugues, whose works, while conveniently located for fertilizer distribution and are yet further away from the source of power, are charged the same tariff.

Under Portugal's Second Six-Year Plan, the nitrogen industry is to play an important part in economic development, the aim being self-sufficiency ultimately in nitrogen fertilizers. In the past the industry has suffered by being dependent on electrolytic hydrogen for ammonia production. Climatic conditions such as a dry summer can almost halve nitrogen production capacity. That this is so is clearly indicated when production for 1957 is considered, which was noted for its outstandingly dry summer, particularly in Portugal. In the past Portugal has played an important part in the plans of Western European manufacturers whose N fertilizer production was geared to a sizeable volume of exports. Now Portugal plans to meet an increasing proportion of its own needs and, what is more, to supply a part of the requirements of its African dependencies. The favourable location in respect of fertilizer raw material supplies and the country's traditions as a maritime trading nation, holds out prospects for its nitrogen industry playing an important part among world suppliers.



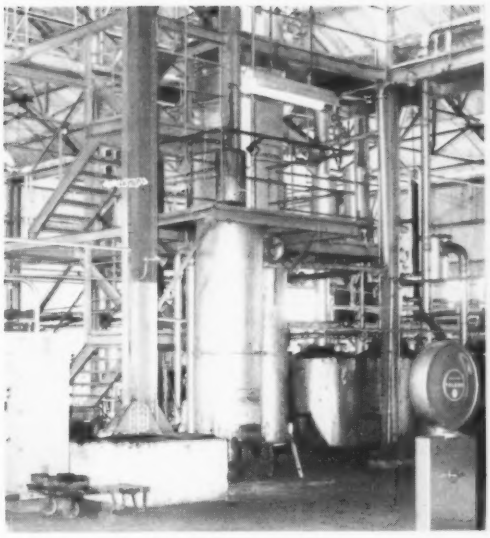
Part of Soc. Montecatini's Ferrara nitrogen plant

Nitrogenous Fertilizer Production at the Integrated Lignite Project for Neyveli

IT was in the late '30's that attention was focused on the occurrence of lignite in and around Neyveli when wells were being drilled for water supply purposes. Geological surveys between the years 1943 and 1951 proved the existence of reserves of lignite, estimated at 2,000 million tons over an area of 100 square miles and at a depth of 180ft. below ground level.

With the urgent need for alternative sources of fuel in Madras State and India as a whole, both for generation of electrical power and for use in industries and domestic purposes, it was considered that the lignite deposits in Neyveli might be the answer to the power problems and could also be the raw material for production of nitrogenous fertilizers.

Under the Columbo Plan, the Government of India arranged for the services of Powell Duffryn Technical Services Ltd., to prepare a report on the economic mining processing and utilisation of the lignite, and after this was received in 1954 and studied, the Government of India took over financial responsibility for the project from January 1955 and the administrative responsibility from September 1955. In November 1956, a Government private limited company—the Neyveli Lignite Corporation Ltd., was formed to control and manage the project. Consultants for the mining scheme are Powell Duffryn Technical Services Ltd.



The pilot carbonisation plant

It is envisaged that under the Neyveli integrated lignite project 3½ million tons of lignite a year will be mined by opencast mechanised methods which will be utilised to produce 250,000 kW thermal power, to manufacture 152,000 tons of nitrogenous fertilizer in the form of prilled urea (equivalent to 70,000 tons of plant food nitrogen), and to process the remaining lignite to yield 380,000 tons annually of carbonised briquettes for use as domestic fuel. During manufacture of the latter, 43,000 tons of char dust, 6,400 tons motor spirit, 51,300 tons of tar and 1,032 tons of phenols will be obtained as by-products.

The Integrated Lignite Project at Neyveli envisages:—

- **Mining of 3½ million tonnes of lignite a year by opencast mechanised methods.**
- **Utilisation of the lignite to produce 250,000 K.W. thermal power.**
- **Construction of India's second largest fertilizer plant which manufactures 152,000 tonnes of nitrogenous fertilizer in the form of prilled urea (equivalent to 70,000 tonnes of plant food nitrogen) annually.**
- **Processing the remaining lignite to yield annually 380,000 tonnes of carbonised briquettes for use as domestic fuel.**
- **By-products from the carbonised briquettes—43,000 tonnes char dust, 6,400 tonnes motor spirit, 51,000 tonnes tar and 1,032 tonnes phenols.**
- **Overburden deposits of fire clay and china clay will be mined.**

Also, in mining the lignite, the overburden containing deposits of fire clay and china clay will be mined. The china clay will be washed and marketed. Overall cost of the project is estimated at about Rs. 86 crores (£64½ million).

Lignite Mining

Underground mining of the lignite proved impossible as the artesian water below the lignite bed exerts an upward thrust of 6.6 to 8.8 kilogramme per sq. cm. and there is no impermeable barrier of sufficient thickness below the lignite bed to withstand the pressure of the water.

Water Control. A definite scheme has been drawn up to control the pressure of the artesian water and eliminate risks of flooding. Some 48 wells will be in operation giving 48,000 gallons of water from the artesian acquifers. This water, the only source available, will meet the requirements of the power station, the fertilizer factory, the briquetting and carbonising plant, the clay-washing plant and the town of about 9,000 houses. A reservoir for storing the water has been constructed and surplus water will be made available for irrigation.

Mining. The mining scheme consists of two phases, the development phase and the production phase. During the development phase, expected to be completed in 1961, the first mine cut will be worked, and adequate quantities of lignite will be exposed. Mining will follow the pattern adopted in Germany and Australia for opencast lignite mines.

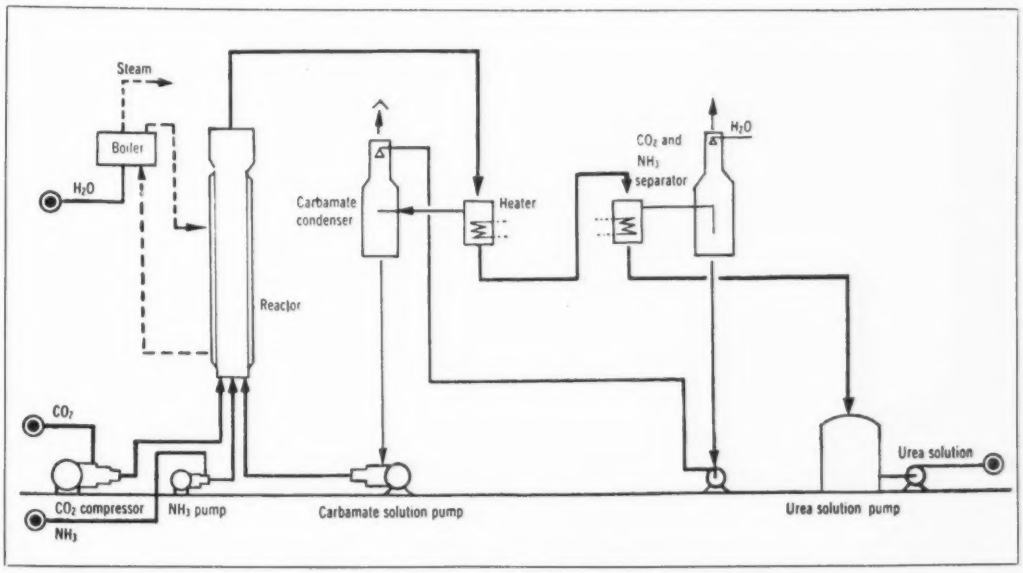
The Power Station

The scheme for utilisation of about 1½ million tons of lignite a year for generation of 200,000 to 250,000 kW of electrical power has been drawn up by the Central Water and Power Commission. It will be financed out of 500 million roubles credit offered by the U.S.S.R. Equipment and machinery will be supplied by Technoexport, Moscow. The first unit of the power station is scheduled to be in operation in June 1961, and the whole power station is to be working at full capacity by the middle of 1962. Provision is being made for a sixth turbine so that installed capacity can be raised to 300,000 kW.

Fertilizer Production

The scheme involves production of fertilizers with a fixed nitrogen content of 70,000 tons a year in the form of urea (152,000 tons) using the Montecatini-Fauser total recycle process. The $52.5 million fertilizer plant is now under construction, and, when in full

General view of the Neyveli lignite mines



Flow diagram of the Fauser-Montecatini recycle urea process

production by the end of 1962, is expected to consume about 35,000 kW of power, 8 million gallons of water a day, and about 0.8 million tons of lignite a year. About 500,000 tons of lignite a year will be gasified by means of oxygen and steam to yield synthesis gas, which will, after purification, be converted to ammonia. The carbon dioxide which is removed in the purification step, and the ammonia, will be combined to produce urea.

In the gasification plant, lignite will be prepared, dried and gasified using oxygen and steam. Three gas generators with a total daily capacity of 685,000 Nm^3 of $CO + H_2$ will be used. The gases, after cooling and cleaning, will be sent to a gasholder.

In the gas purification plant, the gases will be compressed to about 15 atm. and then purified hydrogen sulphide being removed by the Alkazid scrubbing process. This will be followed by carbon monoxide conversion, whereby most of the carbon monoxide will be transformed into carbon dioxide by passing the gas mixed with steam over a catalyst at 500°C. The gas stream after cooling and removal of final traces of sulphur will be treated in a potash scrubbing system to remove the carbon dioxide which will be regenerated subsequently by a caustic soda wash. The purified gases consisting mostly of hydrogen and traces of carbon monoxide and methane will enter the gas fractionation plant. Here, at very low temperatures, the impurities will be partly condensed out and washed out with liquid nitrogen so that pure hydrogen becomes available for synthesis ammonia. An air fractionation plant will supply oxygen of 98% purity for the gasification of lignite and pure nitrogen for use in the gas fractionation step.

Ammonia Synthesis Plant

In this plant, which will use the Fauser-Montecatini process, the pure hydrogen-nitrogen mixture will be compressed to about 300 atm. and then sent through a converter containing reduced iron catalyst at a temperature of about 500°C. The ammonia formed will be condensed by cooling and the unconverted gas will be recirculated back into the converter through an ejector with the help of fresh compressed synthesis gas feed. The daily production of ammonia, which will be 285 tonnes, will be stored as liquid in two Horton spheres.

Urea Plant

The liquid ammonia will be pumped to the urea plant which is planned to date

(152,000 tonnes a year), will be the world's largest. The ammonia and carbon dioxide gas compressed to about 200 atm. will be pumped into a reactor lined with special alloy steel. In this reactor ammonia and carbon dioxide will combine to form urea and water at a pressure of 200 atm. and a temperature of 180°C. The unconverted ammonia and carbon dioxide will be separated from the urea solution, after reduction of the pressure in two stages.

Unconverted ammonia and carbon dioxide gases will be scrubbed with water in two stages and the solution of ammonium carbamate obtained will be pumped to the reactor with the fresh ammonia and carbon dioxide feed.

Urea solution will be concentrated in evaporators to about 98% and the urea melt will be sprayed from the top of a prilling tower. The urea prills of 1 to 2 mm. diameter from the bottom of the prilling tower will be dried and sent either to storage or to a packing station for bagging into 50 kg. Alkathene-lined jute bags. Daily capacity of the plant will be 465 tons of prilled urea. Provision has also been made for the manufacture of 50 tons a day of technical grade urea for use in plastics and other industries.

An important feature of the Fauser-Montecatini process is the use of a pumped carbamate recycle stream. By this means a large proportion of the unreacted ammonia and carbon dioxide in the reactor effluent is recycled to the reactor as an aqueous solution of ammonium carbamate. This development represents a substantial improvement over previous practice in which it was necessary for all of the unreacted ammonium and carbon dioxide to be separated and recompressed. Also operating and initial investment costs are substantially reduced for that portion of the recycle which is carried out as a liquid solution rather than as vapour. In carrying out the most up-to-date arrangement of total recycle in liquid phase, the residual gases, separated under low pressure from the solution after the first recycle, are also recycled as a liquid solution to the reactor. This is carried out by means of a second recycle section where gases separated from the solution and condensed are recycled to the first condenser. This liquid phase recycle allows reduction in power and steam consumption as well as in the investment cost in comparison with the residual gas selective separation arrangement. Elimination of the use of equipment for selective separation of residual gases is slightly less efficient but the liquid phase recycle arrangement affords considerably greater simplicity of operation coupled with lower operating costs.

A major problem in urea plants has been corrosion. At Neyveli, using the mild and carefully controlled reactor conditions laid down by Fauser-Montecatini, corrosion will be prevented. By careful selection of composition and temperature in the reactor, commercially available stainless steel can be used for the reactor lining and other surfaces exposed to corrosion. Silver and lead are not required while initial reactor fabrication has been

Pilot carbonising and briquetting plant under erection

simplified to ensure extended trouble-free operation.

Ancillary Plant

Included in the Neyveli fertilizer plant will be the steam-producing installation to meet the factory's steam needs, electric power distribution system, and off-site facilities such as workshops, cooling towers, laboratories, etc. The steam installation will burn lignite and generate steam at about 40 atm., reducing this to the desired pressures for the different sections of the plant by means of a turbo-generator. Power for the fertilizer plant will be drawn from the nearby power station, while the water for cooling, boiler feed system and process purposes will be taken from the reservoir of artesian water. The fertilizer unit will, if necessary, also supply the hydrogen requirements of the tar hydrogenation unit in the briquetting and carbonising plant to make synthetic fuel.

At present, preparatory investigations are in progress in a modest laboratory attached to the Neyveli fertilizer plant on problems of water treatment, storage of urea, corrosion tests, etc.

Contracts for the f.o.b. supply, erection, start-up and commissioning period of the fertilizer unit were signed on the 27 October 1959 with Pintsch-Bamag AG., with Linde AG. of West Germany for the Winkler gasification, gas purification and air and gas fractionation plants, and with Ansaldo of Italy for the ammonia, and urea plants, steam installation, electric installation and off-site facilities. Site clearance and construction work on the fertilizer works has begun.

Summary

When completed, the Neyveli fertilizer plant will incorporate many innovations. Thus, it will be the first plant in India to—gasify lignite or any other fuel using oxygen and steam; use an ejector system in the ammonia synthesis plant, and manufacture urea by the total recycle process.

Consumption of urea in India is now at an annual level of 30,000 tons of nitrogen and while the Neyveli plant, together with those at Sindri and Trombay will be able to meet a demand four times greater than the above, it can reasonably be expected that urea requirements will rise to 220,000 tons of nitrogen by 1965.

As such the Neyveli project will be one of the more important of the eight fertilizer plants now being erected in India. It is understood also that in deciding the types of fertilizer to be manufactured at these various plants, care has been taken to meet localised demands for certain fertilizers.

With regard to the production of the fertilizers produced at Neyveli, no definite costing has yet been published. In their assessment of costs for urea, the India Government will undoubtedly bear in mind the figure of £100 a ton N for imported fertilizers.

It is unlikely, however, that the cost of making N from lignite will be greater than the cost of imported material. For the other major fertilizer works now under construction in India—Sindri, Nangal, Trombay—the production cost per ton of nitrogen is respectively £86, £65 and £59.

I.C.I.'s Ardeer Nitric Acid Plant now Completed

Second Concentration Plant Under Construction

THE skyline at Imperial Chemical Industries' Ardeer Division's plant has changed with completion of the new nitric acid plant. The group of six absorption columns which is a prominent feature of the new plant is illustrated on p.12. The new plant is now being commissioned. It will produce dilute nitric acid and will use intermediate oxidation pressure plant. Plant for concentrating dilute nitric acid by the magnesium nitrate process is due to be completed in 1961.

The intermediate oxidation system is of a type that has proved its worth on the Continent. The new plant will replace I.C.I.'s existing Ardeer nitric acid plants, two of which are of the Dupont pressure oxidation type, the third being an atmospheric pressure oxidation unit.

The new plant will have greatly improved efficiency as a means of transforming ammonia into nitric acid and will combine the best of both high and low pressure systems. Conversion of ammonia to oxides of nitrogen will be at atmospheric pressure, but when the gases pass to the absorption towers they will be at a higher pressure.

A feature of the plant will be low running costs. It will be almost self-sufficient in the matter of energy, heat released in the process being used to raise steam which, passed through turbines, will operate the plant compressor.

Three large 9ft. converters will use platinum rhodium gauge as catalyst. Beneath each will be a boiler that will use the heat of process gases to raise steam (about 8,000lb. of steam at 270 p.s.i.) to operate a turbine driving a 2,000-h.p. stainless steel centrifugal six-stage compressor. This will raise the pressure of the reaction gases to 42 p.s.i.; more heat will be extracted and transformed into power, using heat exchangers. The gases will then pass to six stainless steel absorption towers, each 60ft. high and 8ft. in diameter. The acid for storage will be of 60% concentration. The bulk of this acid will be used in the division's new magnesium nitrate-type concentration plants.

The group of absorption columns at I.C.I.'s nitric acid plant

Structural steelwork of the second unit to concentrate dilute nitric acid has reached its 110ft. height and equipment is to be installed shortly. This unit resembles the first which is reported by I.C.I. to have amply fulfilled expectations. Capacity of the new plant will be 16,000 tons of 99.5% nitric acid a year.

In the process used to concentrate nitric acid, a strong solution of magnesium nitrate in water is used instead of sulphuric acid. The process has been developed by I.C.I. from work begun in the U.S. I.C.I. Nobel Division carried out the development work that has resulted in the design and construction of the first plant. Magnesium nitrate solution, 72% strength takes up water from dilute nitric acid of 60% strength. The resulting nitric acid has a 99.5% concentration.

A series of interconnected column stripping columns, condensers, coolers and absorption column, and a system of reconcentrating the magnesium is kept in careful balance and control. For this plant equipment special construction materials have been used to cope with the corrosive action of hot nitric acid solution. For several items of equipment high silicon iron has been used and gaskets, joints, valve seats and small-bore pipelines are made from Fluon (polytetrafluoroethylene). In some sections of this second unit, I.C.I. titanium is being used. Area occupied by the plant is compact, totalling a base area of some 40 sq. ft.

The new unit is required for the division's new nitric acid plant described above. Ardeer uses the nitric acid for production of nitroglycerine, nitrocellulose, ammonium nitrate, potassium nitrate (and magnesium nitrate for the concentration units), isopropyl nitrate and other nitro compounds.

Supplies - Prices - Trends

Steady Expansion of Nitrogen Supplies

WORLD supplies of nitrogen continued expanding steadily during the closing months of the fertilizer year 1959/60 and it is estimated that available supplies at 11.3 million tons N were nearly 9% greater than in the preceding year. World consumption appears to have increased at a slightly higher rate so that apparent stocks should show a small decrease. To the industry these results should afford a large measure of satisfaction. At this time last year there was much concern, notably on the part of European producers, over the rapid growth of new plant capacity and the level of nitrogen production, which held a threat to the already low level of prices. West German nitrogen producers in particular were warning that the world-wide additions to nitrogen production capacities were not justified. Instead, from both sides of the Atlantic the indications are that demand in the fertilizer year 1959-60 had kept pace with the record output and for the first time in the industry's history consumption has exceeded 10 million tons N.

In consequence the tone of the market has become firmer but despite this trend there have not been any significant advances in nitrogen fertilizer price in export markets. Instances of exceptionally low quotations are, however, becoming fewer, partly due to the shortage of spot supplies among West European exporters and also as the result of determined efforts by the major exporters to reverse the decline of prices of the past three years. Perhaps the most significant feature of the past months has been the gradual disappearance of the wide price discrepancies in different overseas markets.

Domestic fertilizer prices, which in seven European countries still enjoy the support of some form of Government subsidy, are being further reduced, but in general their level remains substantially above export prices, partly on account of the continuance of high import tariffs in most major producing countries.

Although in general the supply picture is at present balanced, there are a number of producers in Western Europe who appear uneasy at the slow rate of disposal of their seasonal build-up of stocks. This trend should become very much clearer, however, during the next two months when the full extent of purchases by China has become evident. A disturbing feature to suppliers of ammonium sulphate is the appearance on the market of low-priced U.S. by-product material which has failed to find a domestic outlet. The impact of Japanese nitrogen fertilizer supplies, particularly of urea, on world export markets also causes uncertainty, as despite widespread enquiries no substantial sales appear to be made. The overall stock position in Japan, however, is much improved and can be considered virtually down to normal levels.

The outlook for the fertilizer year 1960/61 is promising. Nitrogen fertilizer demand remains buoyant and especially in underdeveloped countries it is being boosted by Governmental and international action, notably F.A.O.'s Freedom from Hunger Campaign. Some 350,000 tonnes N of new capacity is expected to become available during the coming year, but to meet the anticipated higher level of consumption—estimated 11½ million tonnes—existing plants will be called on to produce more. Although this will result in a greater employment of installed capacity there is still a substantial excess which, coupled with the known plans for new plant installation notably in India and Latin America, will continue to exercise a restraint on the level of prices and impose on the major producers the anxieties of intensified competition.

UNITED STATES

Fertilizer output and sales up to the end of 1959 reached record levels in the U.S.A. with sales estimated at no less than $900 million and possibly more. But while the total

value of fertilizer sales is higher because of greater tonnages delivered, average prices per ton paid by the farmer were less than in 1958; prices of nitrogenous fertilizers in particular were 3% lower last year. According to recent estimates nitrogen fertilizer supplies in 1959-60 will reach 3,071,000 tons N, an increase of approximately 11% over the previous record quantity for 1958/59. By March nitrogen producers felt that the long spell of unfavourable weather conditions would cost them business. Although the lateness of the season might result in lower fertilizer usage in order to get the crops started this may be compensated by more top dressings later in the year. Sustained demand resulted in heavy fertilizer movement in April. Supplies of all nitrogenous fertilizers were adequate, except for anhydrous ammonia which was in short supply in several regions. Following the rapid improvement in ammonium sulphate supplies after the steel strike, a surplus has developed recently at several major by-product works. Present ammonium sulphate capacity is 5% greater than last year; at the same time sales in South and South-West have slowed and the late winter caused loss of markets in the prairies and states.

Production of fertilizer grade ammonium nitrate continues to rise, providing increased supplies of solid ammonium nitrate, of ammonium nitrate solution and of lime ammonium nitrate. Ammonium phosphate output is expected to be at a record level in the fertilizer year 1959-60 and urea requirements, particularly for the West Coast, continue to advance. Domestic urea production is at present at an annual rate of 600,000 short tons, almost 75,000 tons greater than last year. Whereas exports continue to decline and should prove to be less than one-half of last year's total of 86,000 tons, imports are rising. Total installed capacity at the end of June is estimated at 840,000 tons to be augmented by some 73,000 tons before the end of the year. Nitric acid demand for fertilizer use reached its peak during March. Heavy movements to other industries has been evident in April and May.

Despite the concern about overcapacity of anhydrous ammonia, Solar Nitrogen Chemicals Inc. have started construction of a $15 million plant near Joplin, Mo., which will be producing substantial quantities of the popular fertilizers as well as urea and related products. The ammonia unit is expected to be completed in about one year. Reasons for the new plant advanced by Solar Nitrogen Chemicals are the increasing consumption of anhydrous ammonia by the two parent companies—Atlas Powder Company and Standard Oil Company (Ohio)—and the establishment of an improved service to customers.

Calspray's (California Spray-Chemical Corporation) nitric acid and fertilizer complex came into production at Kennewick, Washington, on 1 April. Output of 150 tons a day of nitric acid will be used in the manufacture of nitrophosphates and ammonium nitrate at a daily rate of 150 tons in the form of prills. On stream also is the $1.25 million nitric acid plant of Co-operative Farm Chemicals at Lawrence, Kansas.

Formally opened last month was Olin Mathieson's " high-analysis " fertilizer plant at Pasadena, Texas, which has a capacity of more than one ton a minute of pelletised ammonium phosphate fertilizer representing a 40% increase over former output. With the new facility Olin Mathieson are adding new grades to their Ammo-Phos line, nitrogen content ranging from 7% to 16% N.

The Collier Carbon Co. will not now make ammonium sulphate at Wilmington; both ammonia and sulphuric acid are to be sold to Filtrol Corporation for its manufacture at Vernon and marketing only will be retained. Filtrol Corporation, a prominent catalyst manufacturer, previously co-operated with Shell Chemical Company's Ammonia Division.

Hercules Powder Company have plans for a 11,000 tons a year urea-formaldehyde unit at Hercules, California, and Allied Chemicals are increasing urea-formaldehyde capacity.

UNITED KINGDOM

Weather conditions having remained favourable, particularly during the second half of the fertilizer year 1959/60, and more fertilizer has been sold in the U.K. than ever before in this period.

With the increasing demand for nitrogen

fertilizers and nitrogen for industrial use in mind, Imperial Chemical Industries Ltd. are making large additions to their existing plants at Billingham—the Haverton Hill unit has been increased by 50%—Heysham, and Ardeer for making ammonia and for making products from ammonia. A new ammonia plant, with a capacity of 100,000 tons a year, and associated plants to make urea and fertilizers will be built at I.C.I.'s new Severnside Works in South Gloucestershire, at a cost of over £10 million. These plants are scheduled to be in operation during 1963 and will compare favourably with any other plant complex in the world. The new plant will be the responsibility of the Billingham Division, whose fertilizer product sales account for about two-thirds of the division's total sales and which is intended to remain I.C.I.'s main nitrogen fertilizer plant. About 60% of I.C.I.'s nitrogen sold now is in compounds such as CCF (concentrated complete fertilizer) and in conformity with this trend, I.C.I. Billingham Division is gradually changing its production pattern so that more of its output is in the form of compounds—urea and complex fertilizers.

In order to meet the rapid expansion of the market in Northern Ireland I.C.I. is building an ammonium phosphate plant at Belfast due to be producing by the most modern methods by 1962. The choice of a plant in Ireland has resulted from the high cost of transporting fertilizers from Billingham.

Use of industrial nitrogen in the U.K. is soaring and is now estimated to total 150,000 tons N. To meet the rising demand I.C.I. may soon extend their Cassel Works' cyanide plant. Last year's output figures at this plant show that average weekly production throughout the year was slightly above the plant's rated capacity. So great has been the increase in demand that last year the plant made as much in a month as it did in a year at the beginning of the century. Cyanide is, of course, being used in production of acrylonitrile, at I.C.I.'s new acrylonitrile plant at Cassel Works, which began production last December. At Ardeer I.C.I.'s new nitric acid plant is virtually complete and is expected to be in operation in June. (See page 11.)

WESTERN EUROPE

All major nitrogen producers on the Continent have reported higher levels of production in recent months. In West Germany strong domestic demand has been supported by increased exports whereas the previous year 1958/9 exports declined 39,000 tonnes N*. During the current year this setback has been made good and the year's total is likely to exceed the 1957/8 record year by about 5%. Rising output has originated wholly from synthetic ammonia plants as by-product ammonia manufacture—100,000 tonnes N in 1959—continues to decline.

In Italy the extension to the A.N.I.C. works in Ravenna is nearing completion and urea production has already started at an annual rate of 50,000 tonnes. Further additions to Italy's ammonia capacity are envisaged by E.N.I. in conjunction with their petrochemical project at Gela, in Sicily.

In France nitrogen output and capacity are increasing rapidly. In 1959 production totalled 667,000 tonnes N—12% more than in the preceding year—and this trend is expected to continue. Operations by Azo Lacq, near Pau in South-West France, where ammonia manufacture is being based on natural gas, lime ammonium nitrate production is scheduled to start in the early autumn.

In Spain domestic supplies still represent only about one-fifth of demand of 300,000 tonnes N. A new state-owned chemical fertilizer plant has begun production in Spain's industrial area at Puertollano, Province of Ciudad Real. Ammonium and calcium nitrate as well as ammonium sulphate are produced and annual output is expected to reach 80,000 tonnes. It is estimated that within four years Spanish production of fertilizers will reach 1,475,000 tons. This should cover the country's demand and save about $45 million a year on imports.

Lengthy negotiations between the Danish and Norwegian interests for a fertilizer project in Grenaa, Jutland, have resulted in a decision to go ahead with the plant. Hitherto the bulk of Denmark's nitrogen fertilizer requirements

* Note: Not 139,000 tonnes N as erroneously stated in NITROGEN 5, p.2.

have been supplied by Norsk Hydro who are to participate in this development through their Danish distributing subsidiary. Also participating in the new company, A/S Ammonia, are the Danish Svovelsyre og Superfosfaat Fabriken and Dansk Andels—gødings forretning. Grenaa has been chosen in preference to Kalundborg, the locality originally proposed, because of its nearby lime deposits. Should it be decided to build the plant it could be in operation by 1962.

MIDDLE AND FAR EAST

Consumption in these areas, the principal export markets, has continued increasing and is now estimated about 9% above last year's level.

Pakistan's purchase from East Germany augments rising domestic ammonia sulphate supplies; in addition, tenders are invited for 86,000 tons ammonium sulphate and 16,000 tons urea. There is, however, uncertainty as to the Government's ability to dispose of the output of 110,000 tons urea due to be available early next year at the Fenjuganj, in East Pakistan.

The tender purchases by South Korea remain one of the major outlets for Japanese exports. Of the 55,000 tons N purchased in January Japan received about 80%, represented by 40,000 tons urea and 117,305 tons ammonium sulphate. The continued high level of imports by Formosa of Japanese ammonium sulphate and of urea, the latter due to the failure of the Nankong urea plant, make this a very important market for Japanese supplies. Under a recent trade agreement with the Philippines, Japan will supply N fertilizer valued at £½ million in return for tobacco.

From the Government of Sudan the U.S. fertilizer industry received an invitation to make offers for supplying certain fertilizers for next year's growing season. The invitation included 27,000 tonnes of urea which is to be delivered between October 1960 and February 1961. Bids had to be submitted by 24 May.

The Government of India has called for bids totalling 80,000 tons of ammonium sulphate for shipment in two lots—30,000 tons by June 1960 and 50,000 by January-February 1961. Similarly 24,000 tons of urea are wanted in two lots—the June 1960 shipment to be 80,000 tons and the January-February shipment 16,000 tons. A tender has also been issued for 20,000 tons of calcium ammonium nitrate and 20,000 long tons ammonium sulphate nitrate, shipment being called for July-August this year. Offers in both tenders were for 13 June, in anticipation of negotiations starting 15 June 1960.

To purchase nitrogenous fertilizers from world sources, Vietnam has been issued a grant of $1 million by International Co-operation Administration. The authorisation covers contracts entered between the period 10 March and 31 July and provides for delivery by 30 September.

Indonesian estimated fertilizer requirements are reported as 300,000 tons N in 1960, rising to 450,000 tons N in 1961 and 500,000 to 600,000 tons N in 1962.

In Egypt expansion of domestic supplies has not so far seriously reduced imports. The latest plant uses refinery gases from the Shell refinery as raw material for the manufacture of 250,000 tons a year of calcium nitrate by the Aswan calcium nitrate plant of Société Egyptienne d'Angrais et Industries Chimiques (S.A.L.). The new 150-ton-a-day nitric acid plant and ammonia unit of Friedrick Uhde Gmbh. which came into operation last year is being expanded to 260 tonnes a day to raise output to 275,000 tonnes a year of calcium nitrate. An ammonium sulphate unit which will use sulphuric acid based on imported pyrites and later domestic refined and recovered brimstone is also under construction by Chemical Construction Corporation, U.S.A. It will produce 100,000 tonnes ammonium sulphate a year by 1961. Nitric acid production will be 138,000 tonnes a year.

After heavy purchases of shipments earlier this year China is again due to enter the markets in about July and August when about one-third of the expected total of about 280,000 tons N for this year is likely to be purchased.

FREIGHT

Ocean freight rates have declined in recent weeks from the reasonably firm levels registered earlier in the year. Grain shipments, the main support of the tramp market, have declined and transatlantic freights have correspondingly weakened. Trade to the Far East, however, has been brisk, mainly under the stimulus of Japanese shipping needs for grain, scrap and coal. Recent phosphate freight rates from Morocco have emphasised this trend, which is also reflected by the freight rates for fertilizer shipments from Europe to the Far East.

TO	*FROM*		
	Japan	*U.S.A.*	*W. Europe*
China	—	—	60s.6d.-67s.
South Korea	$2.50	$12*	62s.6d.
India	$8	$10*	46s.0d.
Indonesia	—	—	52s.3d.-60s.
South America (Brazil)	—	$ 5.50	—
ditto	—	—	$5.30
South Africa	$7.35	—	36s.3d.

* U.S. flag.

PRICES

The level of world prices has in recent weeks remained substantially unchanged, but the tone of the market has become firmer. In May and April ammonium sulphate sales were concluded at £10 per tonne f.o.b. in bulk, the lowest level yet recorded. In the main the wide discrepancies between price levels in various markets are disappearing and more uniform prices prevail.

The fertilizer year 1960/1 starts with a reasonably firm tone and it is not believed that the relatively slow disposal of ammonium sulphate and lime ammonium nitrate tonnage now experienced by some major European producers will cause any deterioration of prices, especially as they all adhere to a policy of price restraint and controlled production rates. Moreover, Japan's stock position has substantially improved and recent sales indicate that she is no longer pursuing sales at any price.

During the coming months the determining factor is expected to be the price level of the sales to China; this buyer is thought to be awaiting current tender prices, notably those in the case of India and South Korea, which should influence her buying policy. Already in a recent sale China appears to have conceded a slight increase on previous contract prices. And in the current supply position this trend can be expected to be upheld. Japan's ability to affect the orderly disposal of ammonium sulphate and urea stocks remains to be seen and it is unlikely that the surplus by-product ammonium sulphate from the U.S.A. will seriously affect world markets.

The outlook for producers therefore remains cautious but justifiably optimistic.

DOMESTIC PRICES

United States

Current posted prices are as follows :—

	per short ton f.o.b./f.o.r.
Coke oven ammonium sulphate	$ 32 in bulk
Synthetic ammonium sulphate	$ 35 in bulk
Ammonium nitrate	$ 68 in bulk
Nitrogen solutions (40.8% N basis 100%)	$128 in tanks
Lime ammonium nitrate	$ 48 in tanks
Sodium nitrate	$ 48 in tanks

In respect of anhydrous ammonia at present quoted $88 per short ton in tank ex-works in all locations except California, new price schedules have been announced by three leading producers including Allied Chemical Co. As from 1 August the price will be $84 and on 1 October the price will advance to $92. Also subject to alteration is the price for nitrogen solutions which from 1 July will be $2 lower. This new price, $126, will remain in effect until 31 December. As from 1 January, 1961, N solution prices will advance to $132.

In California also, anhydrous ammonia price schedules are to change. Hitherto in Central Valley Best Fertilizer Co. too has been charging $72 per short ton delivered based on a $66 ex-works price. As from 1 August this will be increased by $3 to $69, again on 1 October to $72 and on 1 January, 1961, to $75.

United Kingdom

Following the submission by I.C.I. that anti-dumping legislation should be applied in respect of imports from the Continent of ammonium sulphate, the President of the Board of Trade announced in May that no further action seemed to be warranted as German and Belgian sulphate of ammonia manufacturers had agreed to raise their export

prices to U.K. by £2 10s. a ton. No decision has yet been reached, however, on the application for the removal of the import duty which the Board of Trade has under consideration following a complaint by U.K. small farmers. The delay has been occasioned by hearing in full the opinions of all interested parties.

Current quotations per material delivered to farmers' nearest railway station are :—

	per ton	*subsidy*
Ammonium sulphate (21% N) ...	£18.10.0	£7. 7.3
Nitro-chalk (15.5% N)	£21. 5.0	£9.19.6
Nitro-chalk (21% N)	£24. 0.0	£9.19.6
KayNitro (16% N–16% K_2O) ...	£26.17.6	£7.12.6
Nitra-Shell (23% N)	£26. 5.0	£10.18.6

For the fertilizer year 1960/1 I.C.I. and Fisons have announced price reductions for various fertilizer grades ranging up to £1 per ton.

A new concentrated complex fertilizer was brought on the market by Shell Chemical Company with an analysis of 17% N, 11% P_2O_5, 22% K_2O. This fertilizer, marketed under the name Shell No. 1, will be sold at £37 13s.

Western Germany

Current quotations for material delivered to farmers' nearest railway stations are :—

	D.M. per tonne N February-June
Ammonium sulphate (21% N)	1,190
Ammonium sulphate nitrate (26% N) ...	1,200
Lime ammonium nitrate (20.5%) ...	1,225
Calcium nitrate (15.5% N)	1,360
Calcium nitrate (16% N)	1,400
Urea (46% N)	1,190

Belgium

Current quotations for material delivered to farmers' nearest railway stations are :—

	Belgium francs per 100 kg. March-June
Ammonium nitrate	247
Ammonium sulphate	240
Calcium cyanamide (18%) ...	332
Calcium cyanamide (22%) ...	381

EXPORT MARKETS

Ammonium Sulphate

Market prices range from $28.00–35 per tonne in bulk, the price posted by most Western European producers remains £11 15s. ($32.90) per ton f.o.b. in bulk, and reports indicate recent sales by Japan have been concluded at substantially higher levels.

As the result of continued low-priced ammonium sulphate exports to Spain, the Spanish Government has substantially increased the duty on imports of the now liberalised N fertilizers. The new duty is 15% *ad valorem* plus Pesetas 420 per tonne.

With deferment of the South Korean tender to June or July no new awards have been announced for this market since January; the price ruling at that time* in respect of Japanese ammonium sulphate $39.50-40.50 per tonne f.o.b. and c.i.f. tons based on freight rates of $2.50 in foreign flag ships and $8.50 in U.S. vessels.

The February tender* of the Agricultural Bank of Greece was secured by A.N.I.C. at $40.25 per tonne packed in 100 kg. jute bags.

In a more recent sale to Pakistan, East Germany supplied at $44.27 per tonne c.i.f. in bags.

Ammonium Nitrate

Current quotations range from $56 to $63 per tonne f.o.b. A small quantity (10,000 tons) included in the South Korean tender in January was secured by U.S. suppliers at $78.10 per tonne c.i.f. whereas in February French suppliers sold to Greece at $61.30 per tonne c.i.f. in 50 kg. lined jute bags.

Lime ammonium nitrate in W. Europe is currently quoted at $32-$36 per tonne f.o.b. in bags. Offers to South Korea included material from Italy (SEIFA) and Austria at $33 per tonne.

Calcium Nitrate

There has been no significant change in the price levels since the sale of Norwegian material to Greece in February at $35.50 per tonne c.i.f. packed in jute bags.

Urea

Strong demand for urea has been maintained, but there has not been any significant advance of price.

Since the sale to South Korea of 40,000 tonnes in January of Japanese urea at $84 per tonne f.o.b., and Dutch urea at $83 and U.S. material $86.78, offers from principal suppliers have ranged from $80 to $85.

* See NITROGEN 5, p.17.

Suitability of Sodium Nitrate in Warm-climate Arid Agriculture

NO reason to fear adverse effects from the use of sodium nitrate for warm-climate arid agricultures such as those of India or Egypt is the conclusion of Professor Hugh Nicol, Chemistry Department, West of Scotland Agricultural College, Glasgow, who has recently investigated the matter.*

Doubt about the suitability of sodium nitrate, in particular, has arisen, because it has been alleged that this fertilizer cannot be used safely on the alkaline or saline soils on which arid agriculture is widely practised in sub-tropical and tropical regions. Recent studies show, however, that in all soils sodium nitrate has a high value in maintaining an ionic balance propitious for crops. This property is distinct from, and additional to, the established values of sodium nitrate as a carrier of nitrogen and supplier of sodium in reinforcement or partial replacement of potassium as nutrient.

Soil Management

The question of the effect of sodium on the physical condition of soil has been approached quantitatively by Dr. D. Katakouzinos of the Central Geophysical Laboratory in Athens, who has found that sodium nitrate can be used on the adequately drained nine-tenths of Greek agricultural soils without any danger of injuring their structure or properties. Whether sodium salts are harmful or not depends on two factors—the total amount of sodium salts in the soil and the proportion of sodium to calcium.

In considering the total amount of sodium salts in the soil, management of the soil by drainage, reclamation, etc., has to be considered Coastal soil in Europe, where the difference between high and low tides is usually several yards, receives during flooding by the sea a large quantity of sodium chloride, for example, over 40 tons per hectare, or about 17 tons of sodium even if it is flooded by only 15 cm. of water. Temporary damage is sustained by its structure and its cultivation is out for a few years, until the excess of sodium has been washed out of the soil.

That the soil structure is injured by abnormally large quantities of sodium is no reason, however, for concluding that sodium is inevitably bad for soils. Too little sodium is supplied by a normal dressing of sodium nitrate to have any appreciable effect on any soil. For instance, 200 kg. of nitrate of soda on a hectare† would increase the soil sodium by only 10 mg. per kg. of top soil, i.e., by one part in 100,000 parts by weight. In milli-equivalents (me.) this increase, according to Katakouzinos, is about 0.04 me. per litre of soil extract, an amount too small to have a detectable effect on soils already possessing 10 to 20 me. or more of total cations per litre of extract.

Soil containing a large excess of non-nitrogenous sodium salts may be nearly or quite unadaptable to agriculture. If a saline soil is cropped, however, it needs nitrogen. Hence there is no reason why this nitrogen cannot be provided as nitrate of soda. The nitrate nitrogen can be expected to be wholly effective, since it is not subject to "volatilisation," as are ammonium fertilisers, while the addition of sodium to the soil's large natural stock can be considered negligible.

Proportion of Na to Ca

A second factor in determining whether sodium salts are injurious to a soil is the proportion of Na to Ca. This ratio depends largely on whether relatively insoluble calcium salts, notably calcium carbonate and gypsum, are present naturally in the soil or are added. It will be low in any soil unless that soil naturally has both a high content of soluble salts and a high proportion of sodium among its cations. In a non-saline soil there is usually enough calcium present to offset all sodium supplied in normal dressings of sodium nitrate. If only 2% of calcium carbonate is present this

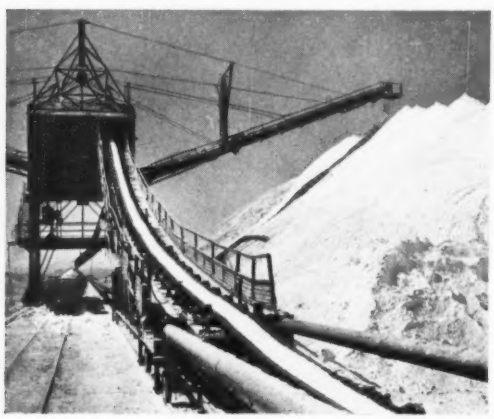
* Chilean Nitrate Agricultural Service.

† 1 Kg. per ha. 0.89 lb./acre. For approximate conversion to lb./acre 10% of the given value in kg. per ha. should be subtracted.

is equivalent to 50 tons per acre (125 tons per hectare) or 20 tons of calcium (50 tons per hectare) in the top few inches alone. A top dressing of nitrate of soda will add only about 0.15 tons of sodium compared with 100 tons or more of calcium for the whole rooting depth. Even if a slight increase in sodium is not offset by calcium supplied in superphosphate or by calcium salts native to the soil an untoward action of sodium nitrate on the structure of well-drained soil need not be feared—even temporarily.

Irrigation Waters and Salts

A much greater amount of sodium can be introduced into soil by irrigation waters which are sometimes highly charged with sodium, than in ordinary dressing of sodium nitrate. For example, the amount of sodium brought to the soil by one acre-foot of irrigation water containing 100 p.p.m. sodium, namely 300 kg., is three times as much as that brought by a dressing of 400 kg. Chilean nitrate per hectare—about 100 kg. sodium—and if, as is usual, "irrigation is applied at the rate of two or three acre-feet a year (6,000 or 9,000 cu. m. water per hectare), the weight of sodium brought by water of quite good quality may be from six to ten or more times as much as all the sodium in the fertilizer." Provided that drainage is fairly good and that the soil is furnished with 5% or more of calcium carbonate and/or calcium sulphate, "not even half a ton or more per hectare of actual sodium (Na) has a bad effect on irrigated soil."



A stockpile of Chilean nitrate

The soil's overall anion-cation weight-ratio should be maintained at about 2.5. If this ratio is lower the supply of calcium or sodium in gypsum, lime, fertilizer or in irrigation water with a ratio higher than 2.5 helps in preserving the condition of the soil. Under actual irrigation, sodium does not accumulate in soil, but if salty water is allowed to stagnate and evaporate it leaves most of its dissolved salts behind as a crust.

Choosing Fertilizer

"Use of ammonium salts instead of sodium nitrate in warm climates can lead to losses of crop and of money spent on fertilizer nitrogen." Such losses are most likely with soils for which sodium nitrate is wrongly considered to be unsuitable, that is, slightly alkaline or calcareous soils. In fact, the only gain in using ammonium salts and urea to sodium nitrate, recent studies by T. N. Jewitt and J. G. Marshall and M. B. Sturgis suggest, is the slight one of making the soil faintly less alkaline. In an alkaline soil ammonium salts and urea decompose first into ammonia, which if it stays in the soil will be transformed into nitric acid. This increase in acidity or decrease in alkalinity is similar to the fractional increase of sodium from an ordinary dressing of sodium nitrate. Urea or ammonia salts cannot therefore be expected to have a beneficial effect on soil pH on calcareous and slightly alkaline soils. These soils, moreover, are precisely those from which, in warm climates, fertilizer nitrogen applied as urea or ammonium salts is likely to escape to atmosphere without giving benefit to soil or crops. Loss of nitrogen as ammonia from sodium nitrate, however, has not been known to occur in practice, and the more calcareous or alkaline a soil in a warm climate the better suited it is by sodium nitrate as a source of nitrogen.

While no nutrient benefit from added sodium can be expected on soils already slightly saline, there is evidence that ammonium nitrogen in warm climates—Spain, Egypt and south-eastern United States—does not come up to expectations as a crop producer while sodium nitrate has been found to give better yields per unit of nitrogen than do ammonium salts.

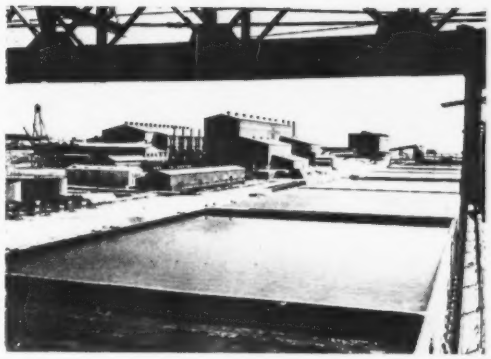
Conclusions

All the available evidence to date indicates that adverse effects from the sodium in sodium nitrate when applied as a fertilizer are unfounded. In fact, irrigation water may often carry greater amounts of sodium compared with that supplied in an ordinary dressing of sodium nitrate.

It is now recognised that sodium nitrate improves ionic balance and therefore should be especially useful on slightly saline soils, since a characteristic of many such soils is a low anion-cation weight-ratio which tends to be unfavourable to growth of most crops.

Another apparently unsound view is that all forms of nitrogen have equal crop-producing effects and hence equal unit value in money. This idea has arisen, it appears, from experience in humid and cool climates, and mostly with acid soils after complete incorporation of ammonium salts in the soil. If ammonium salts are used as top-dressings, the nitrogen in the ammonium salts is of much less value than the nitrate nitrogen of sodium nitrate. This effect has been confirmed by trials in the U.S. and Spain where yields produced by sodium nitrate (16% N) have proved to be broadly indistinguishable from those given by an equal weight of ammonium nitrate (33% total N). Ammonium nitrogen can be equally as profitable and useful as nitrate nitrogen, however, if the whole of it is nitrified. In cool conditions complete nitrification is possible but volatilisation is likely to occur after ammonium salts have been applied to warm, slightly alkaline soil. Hence, the real cost of each unit of nitrogen in urea or ammonium fertilizers may be 10% or 20% higher than the "bought price," while the crop yield is also reduced. These are the conclusions of Professor Nicol.

It will be noted that few references are quoted by Professor Nicol regarding various investigations on the effect of sodium nitrate in warm climate arid agriculture. In the main there is only one reference, and this is from an obscure journal. It is considered, however, that there is probably sufficient information on the use and value of sodium nitrate in warm arid agriculture to make a reasonably authoritative statement on the suitability of sodium nitrate under these conditions, but Professor Nicol has not made use of this information. In particular, it should be noted that the work of Jewitt refers to ammonium sulphate only and not ammonium nitrate, therefore Professor Nicol's use of his results must be deemed irrelevant.



Chilean nitrate extraction plant

In his review Professor Nicol estimates the weight of sodium brought by water compared with the weight of sodium in the fertilizer. This unfortunately is not factual. In support of his view it would have been preferable if some facts had been given as, for example, of the quantity of sodium supplied by rainwater, sodium content of irrigation water, actual consumption of irrigation water per acre, water evaporation, amount of drainage water, the sodium content of drainage water, etc. On such facts a "balance sheet" could have been produced.

Particularly unfortunate is the fact that Professor Nicol has not attempted to deal with what is mentioned in the heading of his paper —the effect of nitrate of soda on physical condition of the soil. Also, throughout the paper the stress is on sodium in sodium nitrate and not sodium nitrate.

A final point is that of ammonium salts and N losses due to volatilisation from calcareous and alkaline soils in any climate, but more particularly in a warm one. Volatilisation can certainly occur, but it should be remembered that losses from nitrate salts take place mainly by leaching and denitrification.

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8th June, 1960.

Half-Yearly Report on the Nitrogen Industry

(all figures in metric tons)

Since our Annual Report issued in December 1959 there has been no cause to make other than minor alterations to our estimates of production and consumption of nitrogen. These have been made in the light of more up-to-date statistics available to us. Estimates for the fertilizer year 1960/61 have been added and show an increase over 1959/60 of 5 per cent in production and consumption of nitrogen for all purposes. Since a number of our readers have raised the point, we feel we should make it clear that our figures for production include the carry forward of stocks from the previous year and might, therefore, more correctly be described as " availability." If this was not the case the additive excess of production over consumption since our reports were first written in 1830 would indeed amount to astronomical figures.

World market prices have again fallen during the last six months and at one time reached, we believe, a rock bottom price for sulphate of ammonia at £10 per metric ton in bulk F.O.B. and trimmed. Recently, however, we note a definite trend towards firmer prices. The stock position is satisfactory and material is not at the moment available in quantity for the export market. An interesting feature about the market is that prices throughout all countries in the world have evened out and no longer are some importing countries paying substantially more than others. This is a healthy sign because in the past prices have leapfrogged downwards as one consumer has seen his neighbour getting a better bargain and therefore determined to do better still himself.

We believe that the logical sequel to the establishment of uniform world market prices is for nitrogen products to be sold on an exchange market as is the case for most other commodities. We shall have more to say on this subject at a later date.

In Spain prices of ammonium sulphate have fallen again and, as forecast in our December Report, this has only resulted in the import duties being raised to protect the home production. The Spanish Government have increased the import duty to 15% *ad valorem* plus pesetas 420 per ton as an anti-dumping measure. If no new cut-price rates are made during the next two or three months, the duty may be restored to its previous level. We estimate that stocks of sulphate of ammonia in Spain at one time amounted to 700,000 tons of product but good seasonal domestic consumption has probably now reduced this figure by half. This heavy stock position arises from the farmers first consuming their own stocks before accepting delivery from the merchants. Before Spain became a full member of the O.E.E.C. and imports were subject to licence, farmers hoarded their fertilizers in the fear that new purchases might be difficult. After liberalisation, when they could buy as they pleased, it was only natural that these farmers should first use up their own purchased stocks. It is for this reason that imports piled up in merchants' warehouses without redelivery to the farms.

In Greece, the Agricultural Bank purchased by tender 40,000 tons of pure nitrogen and, in the form of sulphate of ammonia, the lowest price for material delivered cost and freight in bags was $40.20 and for calcium ammonium nitrate $38.60. A small quantity of nitrate of lime was delivered for the price of $35.25.

In Pakistan, sulphate of ammonia was supplied from East Germany at $44.27 per ton for material in bags on a cost and freight landed basis.

In the Korean tender of 25 January 1960 purchases consisted of 60,600 tons of urea, 12,000 tons of ammonium sulphate nitrate, 1,000 tons of ammonium nitrate and 117,305 tons of ammonium sulphate. Of

these quantities, the Japanese supplied 40,000 tons of urea at prices ranging from $84.000 F.O.B. Japan to $92.50 cost and freight U.S.A. flag liner and all the ammonium sulphate at prices ranging from $39.50 per ton F.O.B. Japan to $48.00 cost and freight U.S.A. flag liner. All this material was packed in bags. The remaining urea was sold by the Americans at $98.48 cost and freight U.S.A. flag liner and by Europeans at $101.50. Ammonium sulphate nitrate was sold from Europe at $52.00 non U.S.A. flag liner and ammonium nitrate from the United States at $78.10 cost and freight U.S.A. flag tramp.

As we write this Report the Indian Authorities have called for tenders to supply 80,000 tons of ammonium sulphate and 24,000 tons of urea. China will be watching this tender closely and any further reduction in price will certainly be reflected in her next season's purchases.

The following are our revised estimates for production and consumption in the world, excluding U.S.S.R., from 1955 to 1961. All figures are in thousand of metric tons of pure nitrogen.

	PRODUCTION (availability)		CONSUMPTION	
	Agriculture	**Industry**	**Agriculture**	**Industry**
1955/56	6,852	1,245	6,327	1,245
1956/57	7,288	1,370	6,997	1,370
1957/58	8,107	1,446	7,660	1,446
1958/59	8,811	1,511	8,358	1,511
1959/60	9,757	1,575	9,273	1,575
1960/61	10,383	1,693	9,812	1,693

The following is the estimated distribution of production and consumption 1955/56 to 1960/61:—

	1955/56		1956/57		1957/58		1958/59		1959/60		1960/61	
	Prod.	Con.	Prod.	Con.	Prod.	Con.	Prod.	Con.	Prod.	Con.	Prod.	Con.
Europe and Egypt	3,853	3,310	4,247	3,635	4,624	3,853	5,078	4,172	5,300	4,400	5,600	4,650
United States	2,700	2,500	2,735	2,735	3,029	2,960	3,100	3,100	3,550	3,500	3,700	3,600
Canada and the Rest of America	298	240	307	260	320	331	340	357	405	409	426	461
Chile	233	17	188	19	194	11	202	11	205	15	205	15
Asia	923	1,299	1,031	1,458	1,279	1,644	1,496	1,902	1,736	2,157	2,005	2,379
Other Countries	90	206	100	260	107	307	106	327	136	367	140	400
Grand Total	8,097	7,572	8,658	8,367	9,553	9,106	10,322	9,869	11,332	10,848	12,076	11,505
Less for Industrial Purposes ...	1,245	1,245	1,370	1,370	1,446	1,446	1,511	1,511	1,575	1,575	1,693	1,693
Total for Fertilizers	6,852	6,327	7,288	6,997	8,107	7,660	8,811	8,358	9,757	9,273	10,383	9,812

CHILEAN NITRATE

During the past year, production in the mechanised plants has continued satisfactorily but in the old Shanks plants has not been maintained owing to high costs and, in fact, a fortnight ago it was said to be almost at a standstill. Since then the Government have stepped in and appointed a new commission to study the situation and technical experts are on the way to Tarapaca to try and work out a new solution. It would appear, therefore, that at last something is being done. Considering the importance to Chile to keep the industry alive in the north, it would appear certain that a solution will be found.

Determining Nitrogen in Fertilizers

Some New Methods Outlined

MOST samples of fertilizers can be satisfactorily analysed for total nitrogen, although not always by the statutory methods. As many of the methods are slow and rather difficult, there is room for improvement. The position regarding determination of the various forms of nitrogen is not so satisfactory, the present trend to high-nitrogen fertilizers and the incorporation of nitrate and/or urea in fertilizers (which may also contain organic matter) complicating analysis.

At the recent Symposium on Fertiliser Analysis held jointly by the Fertiliser Society and the Society for Analytical Chemistry in London in April, H. N. Wilson, Manager, Analytical Group, Billingham Division, Imperial Chemical Industries Ltd., an authority on nitrogen analysis, reviewed comprehensively and in detail a paper on "The Determination of Nitrogen."

In this survey by Wilson, the methods currently available for determining ammoniacal nitrogen, nitrate nitrogen, urea, organic nitrogen total, are critically discussed and attention is drawn to substances that interfere with the determinations; also recommendations are made as to the best methods to use.

Progress in recent years with regard to determination of nitrogen in fertilizers has been very much less than in the case of potassium and phosphorus. The reason for this is that ten to fifteen years ago analytical chemists were dissatisfied with the then available methods, particularly the statutory methods, for potassium and phosphorus, as these were slow and hence expensive and not sufficiently precise. Developments in the physical instruments, however, in the last ten years have resulted in totally new methods for potassium and phosphorus, while new rapid chemical methods have been devised. The situation with nitrogen has been different. Most manufactured fertilizers have contained nitrogen as ammonia or as in lime ammonium nitrate, as ammonia and nitrate. For ammonia-containing fertilizers the universal method for determining nitrogen has been by distillation from a solution containing excess sodium hydroxide. It is not too slow and is one of the most accurate of analytical processes. Nitrate has been determined by reduction to ammonia with Devarda's alloy, which, while more difficult, is not inadequate. Complex fertilizers containing phosphates, potash and nitrogen, other than ammoniacal, were not until recently manufactured on a large scale and statutory methods were good enough for official purposes. Certain mixtures, however, have given rise to difficulty, but as these have been rare less attention has been paid to the determination of nitrogen.

The problem of improving the methods for nitrogen determination has been much more difficult than for potassium or phosphorus. Potassium, however combined, only gives one kind of potassium ion in solution while with phosphates discrimination between those of varying solubility is necessary. The pyro-and also the meta-phosphate which do not give the reactions of orthophosphates, can be simply and quickly converted to the orthophosphate. Nitrogen present in fertilizers in several forms—as ammonium ion, as nitrate, or in various organic combinations, in which proteins (and sometimes their degradation products) predominate, and as urea—is not easily converted quantitatively into one type of compound. Many of the available methods, too, are liable to interference, both by other nitrogen compounds and by other elements present in mixed fertilizers. While most samples can be satisfactorily analysed for total nitrogen, although not always by the statutory methods, analysis is slow and somewhat difficult. So there is room for improvement. When it is desired to determine the various forms of nitrogen, however, the position is not really satisfactory and when high-nitrogen fertilizers and those containing nitrate and/or urea are required to be analysed the position is even less satisfactory.

Ammoniacal Nitrogen Determination

Almost universally a solution of the sample is distilled with an excess of sodium hydroxide solution, the ammonia evolved collected in an excess of standard acid, and the excess back titrated. Wilson's method is to take a sample containing nitrogen equivalent to 40-45 ml. of normal acid, and distil it into 50.0 ml. of normal hydrochloric acid using normal NaOH free from carbon dioxide for the back titration. The indicator is methyl red and hydrochloric acid is used in preference to sulphuric acid as a somewhat sharper end-point is produced.

With this method determination of ammoniacal nitrogen in an ordinary complete fertilizer has a standard deviation of 0.036. This can be improved upon, however, if a calibrated chamber burette is used to measure the acid, corrections are made for departures from the standardisation temperature and the back-titration is made with a micro-burette. The normal acid should always be made exactly 1.000 normal, using sodium carbonate as a standard substance. The sodium hydroxide is standardised against the standard acid, is adjusted to exactly normal strength and protected from atmospheric carbon dioxide.

Wilson is not in favour of standardisation against pure ammonium sulphate because of the difficulty in ensuring that the ammonium sulphate is in fact pure. Also the salt retains moisture even after drying. If necessary, however, suitable ammonium sulphate can be prepared by very slow crystallisation (about 99.95% $(NH_4)_2SO_4$, the remainder being water which can be removed by further grinding and drying).

Apparatus

One defect in determining ammonia is that the usual method is not very quick by present day standards, the time being taken up by the distillation and the necessity of providing an efficient spray-trap to prevent any spray being carried over.

A special apparatus in which the spray-trap is electrically heated so that egress of ammonia is not delayed has been described by Frey[1] and will be considered in more detail in the section on nitrates. Scottish Agricultural Industries Ltd. have used a slightly modified Frey's apparatus for some years for determining ammonia and Dr. I. A. Brownlie of S.A.I. reports that he finds that the distillate contains about 2 p.p.m. of sodium hydroxide—about half the "blank" obtained by Frey, and that at the very longest, a determination of ammonia or nitrate nitrogen takes 20 minutes.

Other Methods

Apart from distillation and titration there are two other feasible methods of determining ammonia in fertilizers, i.e., the old reaction with formalin, and the hypobromite reaction.

Formalin reaction. In the absence of interfering substances this is an often used reaction, although not of the very highest accuracy. It is an official method of the American Association of Official Agricultural Chemists[2] applicable to ammonium nitrate and ammonium sulphate. The procedure is as follows:—

> "After preparation of the solution add about 1 ml. 37% HCHO solution for each 0.1 gm. sample in the aliquot. Dilute to 150-200 ml. and let stand for five minutes. Titrate with 0.25-0.5 N NaOH using five drops of phenolphthalein indicator (if electrometric titration is preferred, titrate to about pH8). Run blank on the HCHO solution."

The end point is quite well defined but Wilson considers potentiometric titration is preferable to the use of phenolphthalein, as hexamethylenetetramine has a slight effect on the indicator. The solution of the ammonium salt must contain no free acid or alkali, nor should it be neutral to phenolphthalein. A pure solution of ammonium sulphate has a pH of about 5.3 and it is to this pH that the solution should be brought. Although ammonium phosphate in the absence of other ammonium salts might be estimated by this method, if the solution were first adjusted to pH 4.5 mixtures containing ammonium sulphate (or nitrate) and phosphate cannot be estimated.

The reactions are:—

1. $(NH_4)_2\,SO_4 \xrightarrow{HCHO} H_2SO_4$
2. $(NH_4)H_2PO_4 \xrightarrow{HCHO} H_3PO_4$
3. $H_3PO_4 + 2NaOH = Na_2HPO_4 + H_2O$ (to pH 8.6)

i.e. in (1) one equivalent of ammoniacal nitrogen liberates one titrable hydrogen ion, but in (2) one equivalent of ammonia liberates two titrable hydrogen ions. If the phosphate content of the

fertilizer is known, the ammonium content may be calculated from the formalin titration.

Hypobromite method. As far as Wilson is aware determination of nitrogen with hypobromite has not so far been applied successfully to fertilizer analysis. It could, however, have considerable potentialities since it avoids distillation, it is rapid and precise and has the added attraction that in principle it could be mechanised and used with a completely automatic titrator such as Brown and Weir's[3]. Wilson, with Dr. E. J. Candlin, has worked out a satisfactory procedure for the reaction but it has not been brought into routine use because it has proved impossible to prepare a reproducible stable hypobromite reagent. Hence, although short-term results are excellent, time spent in re-standardising the reagent, and the possibility of error resulting from decomposition of the reagent has led to its abandonment. If the preparation of a stable hypobromite solution can be achieved, it is considered that the method would find many applications. It could not be used in the presence of urea, however, and it tends to give high results when inorganic matter is present.

NH_3 determination in presence of other N forms. Organic matter and urea interfere with determination of nitrogen. In particular, urea evolves ammonia on heating with alkalis or alkaline solutions and so do some kinds of organic matter. Fish meal, for instance, is liable to evolve aliphatic amines, which are basic enough to be titrated with ammonia. Such interferences lead to high results.

U.K. statutory regulations require distillation with magnesium oxide, but the pH suspension of magnesium oxide is about 11 to 11.5 which is quite high enough to decompose urea and some other types of organic matter.

Another possibility is to precipitate the ammonia with cobaltinitrite, as ammonium sodium cobaltinitrite is almost as insoluble as the better known potassium compound; this process may be used when volatile amines are present. However, it is slow and rather complicated.

The best process for use when urea is present and readily decomposed organic matter is present is the method described by Hamence[4] who considers that distillation under reduced pressure of 40°C is probably the only solution of the problem. Wilson has confirmed that this procedure gives good results.

Table 1
Determination of Ammonium Nitrogen in Presence of Urea

Sample	% urea	% ammoniacal nitrogen	
		Present	Found
B	82	3.0	3.11
C	26	12.5	12.5
D	36	10.9	10.9
E	62	6.4	6.4
F	80	3.2	3.2

In determining ammonia in the presence of urea, the important point is that hydrolysis of urea is negligible below about 40°C and if speed is not important Conway's micro-diffusion technique[5] could well be applied to fertilizer solutions for it is very suitable for dealing with large batches of samples, and accuracy of the method is far greater than that of most biochemical analysis.

Nitrogen as Nitrate

Determination of nitrates is not as satisfactory as that of ammonia since interferences are much more common and more serious. There are in effect two methods available:—

1. Nitrate is reduced to ammonia and determined as such.
2. Nitrate is made to oxidise some reducing reagent and determined either directly or by determining the excess of reducing reagent after the reaction. The methods available include Devarda's, the ferrous sulphate-zinc method of the American Association of Official Agricultural Chemists and reduction with iron powder.

Reduction Methods

Using Devarda's alloy—British statutory methods specify Devarda's for nitrates. Experienced analysts can obtain good results with this method but in less expert hands, it is very erratic.

Details for Devarda's method are somewhat sparse: An aliquot part of the solution is transferred to a flask and a quantity of finely powdered Devarda metal added, which should not be less than six times the weight of the sample present in the aliquot taken. Excess of concentrated alkali shall then be added and the flask at once connected to a distillation apparatus. After standing 30

Table I

Ammoniacal Nitrogen (time per determination 10 minutes)

	Rapid method (1)	Rapid method (2)	Statutory method
Ammonium sulphate	21.04	21.08	21.07
Concentrated complete fertilizer	12.3	12.3	12.2
Ammonium nitrate	17.5	17.5	17.5
" Kaynitro "	13.3	13.4	13.3

Total nitrogen by Devarda's method

	Rapid method (1)	(2)	(3)	(4)	Statutory method
" Nitro-chalk " ..	21.4	21.4	21.3	20.2	21.3
" Kaynitro "	15.6	15.7	15.6	15.1	15.6
Ammonium nitrate ..	34.9	34.9	34.8	32.6	34.8

Time per determination: (1) 25 minutes
(2) 19 minutes
(3) 15 minutes
(4) 10 minutes

minutes to allow the reaction to proceed, heating gently if necessary, the ammonia is distilled into standard acid. Distillation should proceed for at least one hour. Composition of the alloy is arbitrary and the course of the reaction unknown.

There are uncertainties, too, about almost every stage of the reaction, optimum particle size of the alloy, concentration of the alkali and the temperature at which reduction takes place. Hence preference has been expressed for the U.S. method of reduction with ferrous sulphate, zinc dust and sodium hydroxide[6]. M. B. Donald suggests that the alloy should pass through a two-mesh sieve and gives as best conditions:—

No more than 1 g. of nitrate (as $NaNO_3$) 3 g. Devarda alloy, 150 ml. of water and 1.1 to 2.6 g. NaOH (3 to 7 c.c. of 38% NaOH).

B. Dyer and J. H. Hamence[7] for determination of total nitrogen in fertilizers containing organic matter, nitrates and chloride, advocate:—

2 g. sample, 3 g. Devarda alloy, 50 ml. water, 5 ml. 37% NaOH solution (sp. g. 1.4) and a standing period of 30 minutes, with a preliminary digestion period of three hours.

Frey[1], however, says the period of standing is useless and that at the boiling point reduction is complete in two minutes. To prevent the fine spray evolved in Devarda's method, Frey has designed an apparatus in which the distilling flask is surmounted by a metal column about 20 cm. long and 1.3 cm. internal diameter: 15 cm. of this column is packed with metal wool, and to speed up the distillation process, the column is electrically heated up to 120°C. This prevents condensation in the spray-trap with consequent retention of ammonia, so that the distillation is completed in six or eight minutes. Any troublesome frothing is stopped by addition of a minute amount of a fatty oil or silicone anti-frothing agent.

Modification of Frey's apparatus has been carried out by Wilson with substitution of a glass column for the metal one and a few cm. of $\frac{1}{4}$" Dixon rings for his metal wool. Results are stated to be very reproducible and a nitrate nitrogen determination is completed in less than 20 minutes.

Reduction with Ferrous Hydroxide. In this method developed by Cottee and Kahane[8], nitrate is reduced by freshly prepared ferrous hydroxide, the reaction being catalysed by silver. Wilson has used this method for analysis of nitro-chalk for some 12 years.

A sample containing not more than 300 mg. of nitrogen is taken and placed in a 500 c.c. Kjeldahl flask, rinsing the neck down with about 25 c.c. of water. 40 g. AnalaR ferrous sulphate is dissolved in 100 ml. of water and added to the flask, followed by 25 ml. of 0.5% silver sulphate solution. The distillation apparatus is assembled, 25 c.c. of N/1 HCL is put in the recipient, and 100 c.c. of 30% NaOH solution is run into the distilling flask, which is swirled gently to effect mixing. Some 100 c.c. are distilled off in 20 to 30 minutes. Excess acid is titrated with N/1 NaOH using methyl red-methylene blue indicator.

Standard deviation of a total nitrogen determination of nitro-chalk is 0.059 which is but little less precise than determination of ammonia in an ordinary fertilizer mixture. The method is quicker than the statutory version of Devarda's method, and appears to be more easily learnt. The large quantity of ferrous hydroxide present has been said to cause bumping, with the exception of analysis of nitro-chalk. It can be prevented in other cases by adding a gramme or so of precipitated chalk to the flask. Presence of chalk, it has been suggested, leads to a more effective action of the ferrous hydroxide and that quantitative reduction of nitrate is more readily achieved.

Reduction with Iron Powder. This method

developed by Ulsch[9] is being looked upon with increasing favour in the U.S.A. and this version has been described by Ford:—

> 0.5 to 2 g. of sample is placed in an 800 c.c. Kjeldahl flask, add 2 to 5 g. reduced iron (5 g. is sufficient for 0.185 g. NO_3) rinsed with 25 ml. water. Allow to stand to dissolve all salts, then add 25 ml. of cold 1 : 1 H_2SO_4, let stand until visible action ceases and boil 20 minutes to complete reduction. Dilute, add alkali and distil.

In the presence of organic matter Ford used 50 ml. of 1 : 1 H_2SO_4 and to obtain total N content, concludes with a Kjeldahl digestion in the presence of 0.78 mercuric oxide.

Requiring investigation in Ulsch's method is the quality of the reduced iron, for some samples carry excessive amounts of nitrogen presumably as nitride. Satisfactory results have been obtained with ferrum reductum of German and American origin, but a satisfactory English material has not been found.

All the above methods can be applied in the presence of chloride, but not in the presence of urea or some forms of organic matter.

Oxidimetric Determination of Nitrate

One of the oldest methods is the indigo carmine method which is included in the statutory methods as a quantitative test for nitrates, particularly small quantities. For larger amounts there is a group of methods that depend on the reduction of nitrate to NO by ferrous sulphate or chloride in strongly acid solution finishing by measuring the volume of nitric acid produced. This procedure is still an official method in France, Holland, Belgium and Italy. (Gas volumetric methods are not usually very accurate but these do have the advantage of being free from interference by chloride and some forms of organic matter. Urea, however, because of hydrolysis to carbon dioxide would give positive errors.)

Bowman and Scott's Method. This method[11] is not interfered with by urea, but chlorides must be absent, or if present removed by adding silver sulphate to a hot solution of the sample. Up to 2 mg. of sodium chloride in the aliquot titrated can be tolerated. Organic substances that char, or darken with sulphuric acid must, however, be absent. The reaction proceeds in strong sulphuric acid at a temperature below 60°C and preferably at about 15°C for a sharp end point. Recent improvements to the method have been location of the end point electrometrically, one using the "dead-stop" end-point technique[12] and the other a potentiometric technique[13].

Leithe's Method. The reagent used is a solution of 270 g. $FeSO_4.7H_2O$ plus 40 g. NaCl, dissolved in water to which 100 ml. concentrated sulphuric acid is added, and the whole diluted to 1 litre. In the presence of urea sodium chloride (5 g.) should be added. The sample solution is titrated in an acid (H_2SO_4) medium with potassium permanganate solution.

This method[14] has the advantage that it is quick and that neither urea or chlorides interfere. The mechanism of the reaction is obscure, particularly with regard to the added salt, but results are fairly uniformly 97 to 98% of the amount present. An obvious improvement Wilson considers is titration with ceric sulphate instead of permanganate while Belcher and Wilson[15] have described a modification using dichromate for the final titration.

Urea

There is no official method for the determination of nitrogen in urea; it is now rapidly increasing in importance, however. Three methods can be recommended for determination of urea in fertilizers:—

- Urease method.
- *p*.Dimethylaminobenzaldehyde method.
- Sodium nitrite method.

The Urease Method. Application of the method whereby the action of urease on urea converts it to ammonia and carbon dioxide has been described recently by Morgan and Harford[16] in connection with mixed fertilizers containing superphosphate. The results for the range 1 to 10% urea have been very satisfactory (± 0.02%) and the method has the merit that it calls for no "special" apparatus. It has some disadvantages, however, in that not all samples of urease are of satisfactory activity. Secondly urease is inactivated by traces of certain metals including mercury, arsenic and copper. Chromate has a similar effect and even apparatus cleaned with chromate sulphuric

acid and washed can retain enough chromate to vitiate the results. Wilson therefore expresses a preference for the colorimetric *p*.dimethylaminobenzaldehyde method.

Use of p.dimethylaminobenzaldehyde. Described by Watt and Crisp[17] this method depends on the greenish colour produced when *p*.dimethylaminobenzaldehyde reacts with urea in dilute hydrochloric acid. The reaction is not specific, but no constituent of ordinary fertilizers interferes. The colour develops in about 10 minutes and does not fade, and the working range in the final solution is from 40 to 250 p.p.m. urea. In this range the " standard error " is said to be round about 1% of the amount present.

This method has been found satisfactory by Wilson if it is slightly modified. Intensity of the colour is rather sensitive to changes of acidity and care has to be taken to keep this constant. The modified method is as follows:—

10 g. of the sample is extracted in 500 ml. of water by agitation, in a 1,000 c.c. flask, neutralised to methyl red paper, diluted to the mark, mixed, and filtered if necessary. An aliquot, containing about 25 mg. urea, is transferred to a 100 ml. volumetric flask using a burette then 200 c.c. of N.HCl and 20 ml. of the *p*.dimethylaminobenzaldehyde reagent (2.0 g. dissolved in 100 c.c. of 95% ethanol and 10 c.c. of concentrated HCl). This is mixed, cooled, diluted to the mark and mixed again and allowed to stand for 10 minutes. Sufficient of the sample solution is transferred to a 1 cm. cell, and the optical density is measured at 435 mμ. Urea content of the aliquot is read from a calibration graph.

Sodium nitrite method. This has been described by Guelstrom and Demkovich [18] and is based on the reaction

$$2HNO_2 + CO(NH_2)_2 = N_2 + CO_2 + 3H_2O$$

Error at the 95% probability level is less than 2% of the amount of urea present. It has yet to be compared with the colorimetric method under ordinary working conditions. The fact that it does not require a spectrophotometer means that it is suitable for smaller laboratories. Precision appears to be good.

'Organic' Nitrogen

Kjeldahl's method remains the only practical method of dealing with organic nitrogen. Only minor modifications have been reported.

Interferences that arise in Kjeldahl's method are now allowed for in the British Statutory Regulations which specify procedures to be used (a) in the presence of nitrates and (b) in the presence of nitrates and chlorides. In case (a) the Kjeldahl-Jodlbauer method is prescribed; but Wilson advises that conversion of nitrate to nitrophenol should proceed for half an hour in the cold rather than for the 10 minutes specified in the Regulations, and after digestion has cleared to heat for two hours, not one.

Determination of Total N

The statutory method for samples containing nitrates and chlorides follows the lines laid down by Dyer and Hamence[7]. As far as is known, the official methods of no other countries take account of this case, but it is considered that with present-day high nitrogen concentrated fertilizers, the occurrence of chlorides and nitrate in the same sample is likely to become more common.

Urea as a constituent of fertilizers is also becoming more common. Kjedahl's method gives satisfactory results for samples containing up to at least 10% of urea. Urea nitrogen has been determined by Wilson by the *p*.dimethylaminobenzaldehyde method, and ammonia by a method involving the use of ion-exchange resins, in which anions are removed by Amberlite IRA-400 (OH) resin, and cations, including ammonium, by Amberlite IRC-50 (H) resin. Urea is not absorbed and washed away; the ammonium is recovered from the Amberlite IRC-50 (H) by elution with 2N.HCl and determined in the normal way.

Instrumental Methods

The present-day methods of instrumental analysis are not of much use in analysing fertilizers for nitrogen. Thus polarography has been used to a limited extent for determining nitrates, but not in fertilizers; urea is best determined by a spectrophotometric method; and emission spectroscopy and X-ray fluorescence spectrometry cannot be applied to nitrogen. A U.S. company, the Schlumberger Well Surveying Corporation, has made a prototype instrument in which nitrogen in any form can

be fairly quickly determined by neutron activation. The apparatus is quite expensive and very complicated while the results may not be as accurate as those obtained by time-honoured methods.

Conclusions

This recent review by Wilson on nitrogen determination shows clearly that a number of analytical problems still have to be solved. The existence of nitrogen in such a multiplicity of forms suggests that to obtain a general method of nitrogen analysis, it would probably be preferable to go back to an atomic property. The general consensus of opinion regarding what property will be measured and how it will be measured cannot be indicated at this time. There does seem to be some hope, however, in the physical instrumentation methods that are now being investigated in the U.S.A. It must be pointed out, however, that during the last decade there have been in the U.K. only few co-operative investigative studies on determination of nitrogen, particularly as in this country the members of the analytical committee of the Fertiliser Manufacturers' Association have devoted the greater part of their time to analytical methods for fertilizer phosphorus and potassium.

With regard to determination of nitrogen in fertilizers, under the Routine Check Analysis Scheme of the F.M.A., since 1949 some fourteen samples have been tested for nitrogen at different levels. Most of the samples, however, have contained nitrogen only in the form of ammonium salts, but some have contained organic nitrogen and nitrate nitrogen. Another factor is that statistical analysis of the results has been difficult because of lack of uniformity in reporting results from different laboratories.

Preparation of samples containing organic fertilizer ingredients continues to pose problems and statistical samples have shown non-uniformity. The statutory methods of the Fertilisers and Feeding Stuffs (1932) regulations also leave much to be desired especially with reference to exact instructions, i.e., no sample weight is mentioned for any determination; a choice of catalyst is given while the amount of catalyst to be used is left to the analysts' discretion; and, no method of standardising acid and alkali solutions is given and the strength of these solutions is not specified. Perhaps one of the points most emphasised by this review is the lack of an official method for determination of urea which is to-day rapidly increasing in importance. It seems likely that much more investigative work will be carried out on this matter, particularly with regard to the colorimetric *p*.dimethylaminobenzaldehyde and the sodium nitrate methods.

As matters stand at present, much laboratory work has been undertaken on nitrogen determinations but no obvious benefits, either in the form of improved methods or greater precision and accuracy, have resulted, because of non-uniformity in sampling and discrepancy arising because of insufficient detail being given in the statutory methods. The conclusion that must be drawn is that very little further improvement in accuracy or precision of analysis for nitrogen has taken place. The Symposium on Fertiliser Analysis and in particular this paper surveying methods for determination of nitrogen, should do much to stimulate more progress in this matter. At the same time it is to be hoped that the statutory methods of the Fertilisers and Feeding Stuffs (1932) regulations will be reviewed, and amended and revised, so that fertilizer analysts can have suitable foundations on which to base statistical analysis of results and to develop improved or new methods.

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Anti-caking Measures for Ammonium Nitrate

GREAT strides have been made in overcoming caking in fertilizers by granulation techniques. While these have provided a solution in many instances of fertilizers particularly prone to caking such as those with high hygroscopicity, for example, ammonium nitrate, there is still scope for improvement. Apart from special granulation techniques such as that developed by the Dutch State Mines in connection with calcium nitrate, the methods mainly applied have been the addition of anti-caking agents and coating techniques.

In an endeavour to overcome propensity to caking of ammonium nitrate, Paul O. Marti, Jr., Standard Oil Co., Indiana, U.S.A., has developed a particulate ammonium nitrate composition which is substantially non-caking and substantially free-flowing at atmospheric humidity. An improved method of grinding ammonium nitrate in the presence of atmospheric humidity has also been developed which is claimed to be cheaper and quicker than previous methods (U.S. Patent No. 2,901,317).

Copper Oxide Coating

Thus a particulate composition consisting of ammonium nitrate particles coated with a comminuted copper oxide has been produced, the particles of which are characterised by substantially free-flow ability at atmospheric humidity and by a storage stability substantially that of dynamite-grade ammonium nitrate. The copper oxide anti-caking agents may be either anhydrous or may contain small amounts of absorbed water. Either the naturally occurring material or synthetic material may be used while the preferred oxides are: Cupric oxide, CuO, and cuprous oxide, Cu_2O.

The anti-caking agents or grinding aids must have a particle size markedly smaller than the particle size of the ammonium nitrate which is to be treated, and to obtain a substantially complete coating, a comminuted mixture is preferred. Effective agents have a particle size between 0.01 and about 10 microns; as much as 10% by weight having an average size somewhat greater than 10 microns can be present. When the agent is intended for use solely as an anti-caking agent, however, agents having a particle size mainly below 1 micron should be used. The anti-caking agent operates by forming an adherent coating on the outer surface of the ammonium nitrate particle and precluding the water vapour in the atmosphere from contacting the ammonium nitrate. The agent also reduces the agglomerating tendencies of the ammonium nitrate particles so that the coated material flows better than the uncoated particles in a dry atmosphere. The coating does slow somewhat the rate of solution of the nitrate in water.

Finely-divided coated ammonium nitrate particles are obtained by simple mixing of ammonium nitrate particles and a suitable amount of the anti-caking agent. To obtain more finely-divided coated ammonium nitrate particles, nitrate and a suitable amount of anti-caking agent are passed simultaneously through a grinder.

The amount of anti-caking agent required to coat a given quantity of ammonium nitrate particles is dependent upon the size of the ammonium nitrate particles. The smaller the particles the more agent is needed per unit weight of ammonium nitrate. For fertilizer size grains as little as 0.2% by weight, based ammonium nitrate, of cupric oxide or cuprous oxide is sufficient. For dynamite grade ammonium nitrate particles, between about 0.5% and 1% by weight of cupric oxide or cuprous oxide may be required. For the fine powder desirable for some explosive usages where particles are required to pass through a 325 mesh screen, 4 to 5% by weight of cupric oxide or cuprous oxide may be needed.

In general the amount of anti-caking agent needed to obtain a particulate ammonium nitrate composition essentially free-flowing at atmospheric humidity is between about 0.2 and 5% by weight of comminuted anti-caking agent.

One possible disadvantage of use of these anti-caking agents is the catalysis by the agent of decomposition of ammonium nitrate, for the presence of copper oxide in ammonium nitrate can result in an explosion when the nitrate is heated to about 130°C. The higher the purity of ammonium nitrate the more catalyst is required. The amounts of anti-caking agents needed to produce free-flowing ammonium nitrate particles at atmospheric humidity, however, are much less than the amount needed to catalyse the decomposition of ammonium nitrate and, in fact, the coated particles are characterised by storage stability substantially that of dynamite grade ammonium nitrate.

Development of a high-density, dust-free, non-caking prilled ammonium nitrate was announced last year by Monsanto Chemical Co., U.S.A. This product is stated not to cake at a pressure of 600 lb. per sq. inch and on account of its high density requires 20% less storage space than other grades of prilled ammonium nitrate. No details have been revealed by Monsanto regarding the process used. In this instance, however, a coating material is not employed.

Conclusions

Measures to prevent caking are particularly important in the case of ammonium nitrate used as an explosive. Thus considerable care has to be taken to maintain low atmospheric humidity in order to obtain powdered ammonium nitrate of a particle size suitable for explosive use while at present a slow and expensive grinding operation is required to prepare finely powdered ammonium nitrate. This process developed by Standard Oil would therefore seem to hold much promise of more economic and safe manufacture in this instance.

Aerial Distribution of Urea on a Suffolk Farm

ABOVE: The converted lorry

UREA from Norsk Hydro A.B. Norway is being applied by Mr. Frank King to his farmlands at Ramsholt, Suffolk. A new innovation, however, is the use of an aeroplane to distribute this nitrogenous fertilizer.

A lorry has been converted to carry a conveyor-type hopper which is used to load the aeroplane with the urea.

RIGHT: With its load of urea the aeroplane takes off to spread the fertilizer over Mr. King's field

Cyanocarbons, a New Class of Compounds

Give Dyes for Synthetic Hydrophobic Fibres

A NEW class of compounds—the cyanocarbons—has been investigated during the past few years at the E. I. Du Pont de Nemours and Co.'s experimental station, Wilmington, Delaware. At the present time no commercial outlets for these compounds have been established, but it is believed that as more becomes known about the cyanocarbons, they will join hydrocarbons and fluorocarbons as a class of compounds with a wide variety of uses.

Research on the cyanocarbons began after Dr. Theodore L. Cairns of Du Pont Central Research Department speculated that molecules largely or wholly substituted with cyano groups would have an unusual structure and hence interesting chemical properties. Steric considerations indicated that such cyanocarbons could be prepared while electronic theory pointed to an electronic configuration that would confer unusual reactivity. Tetracyanoethylene was the first cyanocarbon prepared and was obtained by a member of Dr. Cairns' research team, Dr. Richard E. Heckert, by treating malonitrile with sulphur monochloride. Later in conjunction with Drs. Ernest L. Little and Rudolph A. Carboni, Dr. Heckert synthesised this cyanocarbon by reacting dibromomalonitrile with copper in boiling benzene. A 55 to 65% yield of pure product is obtained.

$$2\ Br_2\ C\ (CN_2) + 4Cu \longrightarrow (NC_2)\ C = C(CN_2) + 4\ CuBr$$

Tetracyanoethylene is a colourless, crystalline compound that sublimes readily above 120°C, melts at 198° to 200°C, and boils at 223°C. The compound has high thermal stability and can withstand short exposures up to 600°C. Even when sublimed through a tube at 1,000°C, half of it is unaffected; the remainder is cracked to cyanogen and tar.

Tetracyanoethylene

The first cyanocarbon prepared

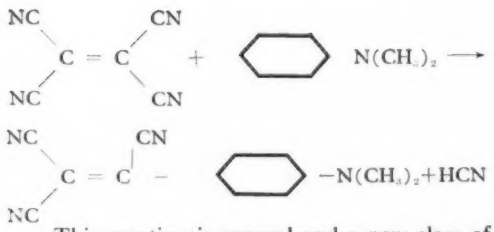
Tetracyanoethylene has a high heat of combustion and a high flame temperature; in particular, it has been found that a tetracyanoethylene-oxygen flame is much hotter than an acetylene-oxygen flame.

Solutions of tetracyanoethylene in aromatic hydrocarbons are intensely coloured—benzene solutions yellow, xylene solutions orange and mesitylene solutions red. This phenomenon has been studied in detail by Dr. William D. Phillips and Dr. Richard E. Merrifield who attribute the colour to formation of a 1:1 complex between tetracyanoethylene and the aromatic hydrocarbon, as a direct consequence of tetracyanoethylene's electronic structure. It is an electron acceptor (Lewis acid) and has a strong affinity for electrons. Since tetracyanoethylene has a double bond deficient in π-electrons it is a special kind of Lewis acid—a π-acid and, in fact, is the strongest π-acid yet reported. Aromatic hydrocarbons are rich in electrons and are Lewis bases (electron donors) and with these, ethers and olefins tetracyanoethylene forms complexes.

Addition reactions take place with hydrogen, sodium bisulphite, and ketones with α-hydrogens. Particularly striking is the addition of tetracyanoethylene to dienes to give Diels-Alder adducts.

With tetracyanoethylene, breaking a C-CN bond occurs with great ease and even exposure to moist air causes it to hydrolyse gradually with evolution of hydrogen cyanide. The other main product of hydrolysis is tricyanovinyl alcohol. This compound has an ionisation constant of about 1.1×10^{-2} in contrast to that of 1.75×10^{-5} for acetic acid. Hence, this cyanocarbon acid's ability to ionise is several hundred times greater than that of acetic acid.

Mixed with dimethylaniline, tetracyanoethylene gives a very bright red solution.



This reaction is general and a new class of dyes has been discovered—the tricyanovinyls. These are intensely coloured as a result of their highly conjugated structure and give brilliant orange, red or blue dyeings on synthetic hydrophobic fibres such as polyethylene terephtalate and polyacrylonitrile. The dyes have not yet been used commercially, but are still under study by Dr. Heckert and his colleagues.

In a two-step process, two cyano groups of tetracyanoethylene can be replaced by dicyanomethylene groups and the product, hexacyanoisobutylene, is a dibasic acid that is completely ionised in aqueous solution. It is one of the strongest organic acids known and is comparable to sulphuric acid in strength.

Tetracyanoethylene and its derivatives provide a means of making a variety of highly substituted heterocycle compounds not readily made in other ways, Du Pont have found. Such an example is 2, 5-diamino-3, 4-dicyanothirophene.

The significance of Du Pont's investigations of cyanocarbons is that with the synthesis of tetracyanoethylene, the first percyanoolefin to be made, a large new area of organic chemistry has been opened up for exploration. With release of Du Pont's work on cyanocarbons, much interest has been aroused both in the U.S.A. and abroad, where independent investigations are well under way. It is therefore expected that much more will be heard regarding this interesting new class of nitrogen-containing compounds. What is more, if the cyanocarbons follow the path of the hydrocarbons and fluorocarbons in commercial aspects, then they can be expected to have a considerable potential. In one aspect alone, as dyes for the newer synthetic fibres, they would be highly valued.

Enzymatic Nitrogen Fixation

MAN-CONTROLLED fixation of nitrogen by enzymes—long-sought by researchers seems likely to become reality following research by E. I. Du Pont de Nemours and Co., Inc., Wilmington, Delaware. Drs. James E. Carnahan, Leonard E. Mortenson, Howard F. Mower and John E. Castle of this company have for the first time duplicated in a test tube the reaction used in the living cell to create nitrogen compounds. The discovery means that biological nitrogen fixation can now be observed in detail and should enable scientists to analyse the enzyme chemistry within the living cell.

Potential practical use envisaged could be either economic duplication of enzymes or artificial stimulation to increase farming efficiency.

Nitrogen, which is available in large quantities in atmosphere (76.22%N by weight in air), can be used by certain low forms of life in this form. The chemical industry now produces some 10 million tons of nitrogen a year or 2% of the nitrogen needed by the world's fertilized crops, obtaining nitrogen from air using very high temperature and pressure.

Key to the fixation is the discovery of a technique by which nitrogen-fixing chemicals can be separated from bacteria, and under artificial stimulation, perform their function outside the bacterial cell. To accomplish this the living bacterial cells are broken open and the enzymes are extracted under carefully controlled conditions to avoid damage to the delicate enzyme system. To provide the necessary energy for the nitrogen-fixing reaction, pyruvic acid is added. The extracted enzymes then carry out the process outside the cell bodies.

The Du Pont researchers are continuing their investigations.

Use of Caprolactam Waste from Polyamide Production as Nitrogen Fertilizer.

LACTUM residues formed during polymerisation of ε-amino-caprolactam which consist of short-chain intermediates unsuitable for reprocessing, contain 10% to 11% N. These wastes contain when fresh, some 2% of sodium hydroxide (NaOH) and are obtained first in liquid form, but harden on cooling and finally become a firm, compact mass. As such, these wastes have been considered as unsuitable for further use; on the contrary, removal of the waste lactams has meant additional expense to the plastics industry. With the need for more nitrogenous fertilizers in mind, investigations have been carried out by W. Jahn and M. Vogt of VEB Thuringisches Kunstfaser-werk 'Wilhelm Pieck' since 1956, using finely ground waste to ascertain whether nitrogen in the residues was available to plants, etc. Experiments were undertaken using grass, meadow fescue (*Fetuca pratensis*). A grass was selected because of its long growth time and having to use a slowly-acting fertilizer. The experiments were divided as follows:—

(a) without nitrogen
(b) 1 g. N as NH_4NO_3
(c) 0.3 g. N as NH_4NO_3
(d) 1 g. N as caprolactam Ia < 0.2 mm. particles
(e) 1 g. N as caprolactam Ia 0.2 to 1.0 mm. particles
(f) 1 g. N as caprolactam Ib < 0.2 mm. particles

Control experiments using no fertilizer material were also done.

It was clear from the experiments that nitrogen was available in the caprolactam wastes as indicated by the increase growth of grass. Using nitrogen fertilizer proper the increase growth can be indicated as 100%. With the caprolactam wastes as nitrogen fertilizer the increased growth was 95%. Best results were obtained when the wastes were finely ground (0.2 mm. particle size).

A further series of experiments were carried out in 1957 by Jahn and Vogt using a caprolactam waste designated caprolactam II. The material was ground very fine (1.0 mm. particle size) and was applied direct to summer barley (*Sorte freya*) as follows:—

(a) without N
(b) 60 kg. N in the form of caprolactam II
(c) 60 kg. N in the form of calcium ammonium nitrate

It was noted that there was noticeable injury using the caprolactam waste. (This was not observed in the first series of experiments). The plant material recovered, however.

It was evident that caprolactam wastes were to a large extent harmful to plants and could destroy them. The cause of the damage was traced to the existence of some 2% NaOH in the waste lactam material, but that damage did not always result required explanation.

In the first series of experiments, the caprolactam wastes had been kept for up to four weeks in a finely ground condition, in small quantities of about 10 g. each exposed to the air. The sodium hydroxide had thus been acted upon by the air so that the carbonate had formed. On application, the caprolactam wastes had a strong alkali effect, but were not corrosive so that no damage was caused to the young plants.

In the field experiments, in which the freshly-ground material was scattered on the young barley, virtually 2% NaOH was also being applied and damage to the young plants was apparent.

Growth of mustard and flax subsequently cultivated after lactam waste had been applied to soil for 16 days prior to sowing has been described as "astonishing." In favoured conditions such as damp soil and temperature it is considered that there is successful interchange with the sodium hydroxide and carbon dioxide or other substances with an acidic effect contained in the soil. Since nitrogen fertilizers — particularly calcium cyanamide — also require to be applied some time before sowing seeds, etc. there can be no bar to the use of caprolactam wastes as nitrogenous fertilizers.

Summary

It is obvious from the foregoing that the caprolactam wastes are mainly harmful to plants on immediate application. The specific cause of noticeable damage to the plants is doubtless to be attributed to the NaOH contained in the caprolactam wastes. The deciding factor for this—whether or not harm is caused—might in the first instance be the extent to which NaOH has intermingled with carbon dioxide or other acid substances, so that at least its corrosive effect is no longer present. This material can be obtained by leaving the finely ground material exposed for a longer period in the air, and thus allowing it to mix with the carbon dioxide in the air; or the application of freshly-ground material should occur two to three weeks before the sowing or planting-out respectively of the plants, so that any NaOH still existing can intermingle favourably with soil substances and be rendered harmless.

Insofar as caprolactam wastes containing NaOH could be used simultaneously as a corrosive for attacking weeds and as N fertilizer, this matter still needs elucidating; theoretically, however, it is an entirely feasible possibility.

The commonly known unfavourable action of NaOH on soil can be discounted, as the substances which enter the soil through the application of caprolactam wastes, are only a fraction of those which appear with the usual fertilizers.

It is important that the following should be observed in the use of caprolactam wastes as fertilizer:—

- Wastes should be ground to a fineness of 2.0 mm.; although not essential, storage in the open air would be desirable to assist carbonation of NaOH with the carbon dioxide in the air.
- An interval of some two weeks between scattering the finely-ground lactam wastes and the sowing or planting out of the young plants should be allowed.
- The lactam wastes can be used in the preparation of composts and artificial manure.

The strong alkali action of the caprolactam wastes can be regarded as a favourable effect in areas where there is strong soil acidity; its application should be especially advantageous therefore on strong acid soil, and in the neutralisation of strong acid waters, as for example in lignite districts; in fertilizing extra acid soils (pH = 2) for the purpose of recultivating; in its use in conjunction with fertilizers which contain pure acid (e.g. strong acid superphosphate.)

Determining Total Nitrogen and Nitrogen Distribution in Fertilizer Solutions

DURING the blending and formulation of high nitrogen fertilizer solutions containing more than one type of fertilizer as a supplier of nitrogen, it is necessary that the concentration of all nitrogen-containing compounds in the solution, as well as the total nitrogen content, be quickly and accurately known. Researchers from the Grand River Chemical Division, Deere and Co., J. D. Slater, J. S. Hill and D. C. Sesso, have recently reported on the determination of total nitrogen and nitrogen distribution in fertilizer solutions. For quick analysis a fairly rapid irease hydrolysis of the urea is done, followed by direct titration of the ammonium carbonate formed. The ammonia due to the urea, and any ammonia originally present, is distilled into saturated boric acid solution. The ammonia found by subsequent titration is corrected for the ammonia from urea. Finally, the sample solution remaining is diluted, treated with Devarda's alloy and again distilled into boric acid solution for titration and estimation of nitrate content. This method can be used for urea-water solutions, and urea-ammonia-ammonium nitrate water solutions. Using a suitable sampling technique, solutions with widely varying concentration ranges can be analysed successfully with a precision and accuracy comparable to other methods.

plant and equipment

PHILIPS' AUTOMATIC PLANT FOR LIQUID NITROGEN

DUE TO THE RARITY of liquids possessing low boiling points, the production of low temperatures is normally achieved by the use of liquefied gas, its absorption of heat on evaporation cooling the material to which it has been applied. And understandably, it is the atmospheric gases, notably oxygen, nitrogen and air itself, that are the most widely used.

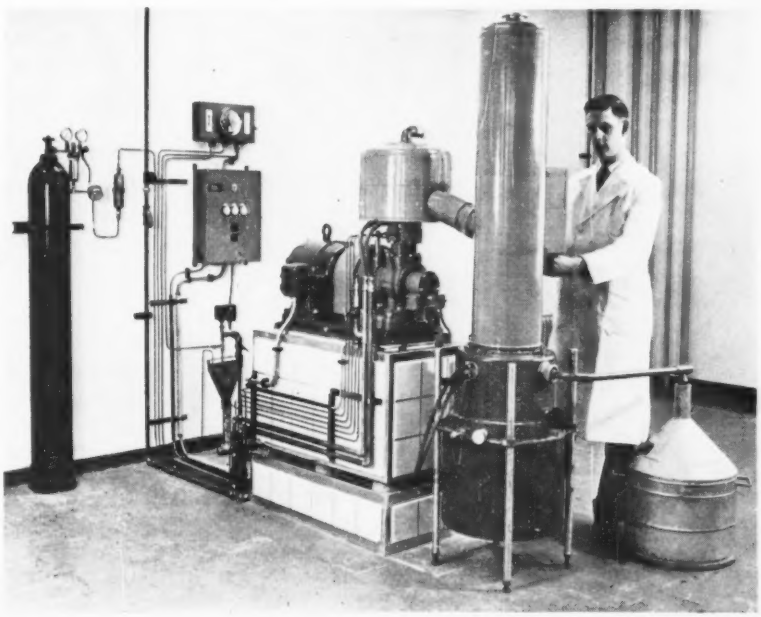
The advantages of using such gases is that they are easy to handle, allow temperatures as low as—196°C to be obtained, whilst in addition the raw material is abundantly available and costs nothing.

In scientific research, particularly in the fields of atomic energy, and solid-state physics, increasing use is being made of liquid nitrogen, both as a cooling medium and in a variety of technical processes.

For many applications nitrogen offers advantages over air and oxygen. Liquid nitrogen, for example, is non-inflammable and is thus to be preferred wherever the presence of oxygen involves a fire risk. Furthermore, liquid nitrogen provides a constant temperature (which liquid air may not) of—196°C, which is 13° lower than that obtained with liquid oxygen.

The Philips' gas liquefier, which is already available as a liquid air producing machine, has now been followed by the development of the Philips' liquid nitrogen plant.†

In this equipment an air-fractionating column, which serves to separate out a portion



Philips' liquid nitrogen installation. After start-up operation is completely automatic enabling the plant to run for periods up to six days without supervision

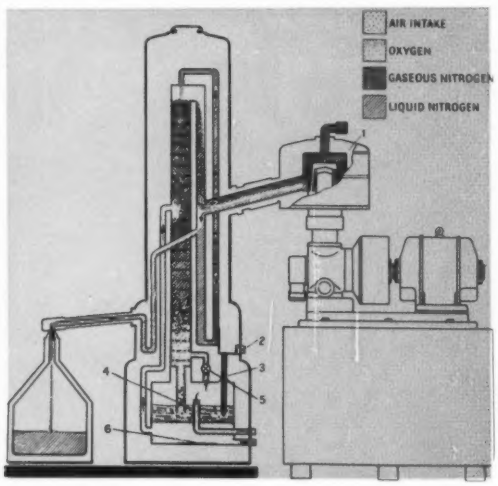
of the nitrogen present in the air, is coupled to a gas liquefier, the latter slightly modified so as to work in combination. Thus instead of air being condensed directly from the atmosphere, the nitrogen fraction is condensed and in consequence may be continuously withdrawn.

The result is a small, simple, automatic plant capable of operating continuously if need be up to six days, over which period it will produce approximately 150 gallons of liquid nitrogen. The gas is not at any stage compressed or chemically purified, and since the possibility of contamination by oil, etc., is completely eliminated, the "end" product has a very high degree of purity.

Construction

The complete installation shows the small size and compactness of the unit.

It comprises the standard gas refrigerating machine with a special condensing head and driving motor on a common baseplate, the whole assembly being mounted on a concrete block to eliminate vibration. The air fractionating column, which stands in its own adjustable supporting cradle, is connected to the condensing head by means of a flexible coupling. The control and instrument panels are arranged for wall mounting. For its operation the plant requires only to be provided with a cylinder of refrigerant gas (hydrogen or helium) for the closed cycle. A Dewar vessel, shown on the extreme right of the illustration, serves as a receptacle for the liquefied nitrogen.



Simplified cross-section of the fractionating column. (1) Liquefier; (2) External channel; (3) Pin; (4) Waste oxygen pipe; (5) Vapour control valve; (6) Electric heater

The total floor space required for the complete equipment, including ample provision for operation and servicing, is 40 sq. ft., with a headroom of 8ft. This small requirement, together with the safety and general quietness of the plant when running, permit it to be accommodated easily inside an existing research laboratory or industrial workshop or, if desired, in a separate outbuilding.

Closed System

The operating principle and construction of the Philips' gas refrigerating machine has been described in various technical papers.*

Briefly, however, the machine operates on a closed system, continuously compressing, cooling and expanding a refrigerant gas (hydrogen or helium). But unlike that in other evaporation refrigerators, the gas does not at any time liquefy. This means that the temperature level is no longer fixed by the boiling point of the refrigerant medium as in conventional equipment, but can instead adjust itself to the conditions under which the machine is operating.

Cold Cycle

The principle lends itself to the construction of a machine of extremely simple design employing two pistons in a single cylinder, an out of phase motion being applied by means of a common crankshaft. The movements of the pistons cause the refrigerant gas to undergo the cycle outlined above, the gas being compressed between them, passed through a cooler to the expansion space, expanded and returned to the compression space, when another cycle commences. A regenerator, acting as a heat store, located between the cooler and expansion chamber, allows the expansion to take place at low temperature.

The gas to be liquefied, in this case nitrogen, is brought into contact with the out-

side surface of the expansion space, where the cold produced causes it to condense.

In distillation, when it is desired to separate one substance from a mixture, the important factor is that there should be a difference between the volatility of the material to be separated, and that of the mixture which will remain.

This is also true for air. When liquefaction takes place the less volatile material, in this case oxygen, tends to condense first, thus leaving the remaining gas richer in nitrogen. On evaporation, the converse takes place, nitrogen evaporating first leaving a liquid richer in oxygen.

Separating Column

This principle is applied in the design of the air fractionating column employed in the Philips liquid nitrogen installation. Arrangements are, however, made firstly, to provide a very large surface area on which the evaporation and condensation can take place within a small volume; secondly, to allow the vapour to pass upwards. This means that the oxygen-rich liquid will, on condensation, drop downwards and, provided the height over which this takes place is sufficient, there will be a continuous removal of oxygen from the vapour as it passes up the column, *resulting in the production of almost pure nitrogen at the top.* This gas is then fed to the gas refrigerating machine, where it condenses, part being drawn off as the liquid nitrogen product and the remainder returned to the top of the column and allowed to trickle down. This results in an overall liquefaction of oxygen which collects at the bottom and may be drawn off as waste.

The design of the Philips air-fractionating column incorporates a completely automatic control system which after start-up allows the plant to run entirely without supervision for up to six days.

Diagram

In order to appreciate the way in which the column and its control system function reference may be made to the simplified cross-section of the column and condensing head.

Atmospheric air is drawn into the column at the base, through a heat exchanger where it is cooled by the oxygen-rich waste product leaving the installation (this warms up the waste gas before it leaves the plant and prevents excessive loss of cold in this stream). In this cooling of the air all the impurities in it are frozen out and condense on the surface of the heat exchanger. At the end of the run they may be removed by defrosting, aided by the electric heater (6), situated in the base.

Vapour Bubble-Pump

The cold, pure, gaseous air now passes upwards and into the column at its mid-point. Here it meets liquid flowing down which, as already described, tends to condense the oxygen, allowing the vapour to continue its passage. As it does so it becomes progressively richer in nitrogen until it leaves the top of the column almost pure. The gas then passes to the gas refrigerating machine where it is condensed and falls back into what may be considered one leg of a U-tube, part of the liquid produced overflowing and passing off through an insulated delivery pipe, into the storage vessel. The other leg of the U-tube is warmed, via a metal strip, by water (2). Vapour bubbles form, which rise, carrying liquid with them into the top of the column. This liquid then trickles down the column, "stripping" the oxygen from the vapour coming up.

Waste oxygen falls to the bottom of the column as liquid and is blown out of the plant through pipe (4), being heat-exchanged with the incoming air on the way. A small control valve (5) serves to regulate the flow of vapour back to the column, this valve being adjusted during starting-up of the plant.

Should too much liquid be carried to the column, resulting in a drop in output, the liquid level at the bottom will rise thus cooling the metal pin (3). This in turn tends to cool the pump, which slows down, reducing the liquid upflow until equilibrium is restored. When too little liquid is carried to the column the pump will work faster until conditions again come under control.

* N. V. Phillips, Eindhoven, Holland, U.K. distributors, Research & Control Instruments Ltd., 207 Kings Road, London, W.C.1.

† Philips Type No. PW7050.

‡ Philips Technical Review, Vol. 16, Nos. 3 & 4, 1954.

current events

INDIA

Durgapur Ammonium Sulphate Plant

On stream is the ammonium sulphate plant at the Durgapur steelworks, designed and engineered and constructed by Simon-Carves Ltd., Cheadle Heath, Cheshire, one of the member companies of the I.S.C.O.N. consortium at Durgapur. The plant utilises by-product ammonia from coking operations for the production of crystalline ammonium sulphate. Rated capacity is 57 tons of ammonium sulphate a day at full output from a gas volume of 2.38 million cu. ft. an hour. Eventually it is intended that Durgapur should produce some 67,500 tons a year of ammonium sulphate, nearly four times as much as the present output.

The crystals are formed in a saturator bath and are then ejected automatically to a continuously discharging centrifuge for preliminary drying. They are fed by conveyor to a rotary hot air dryer and are then automatically weighed into bags ready for sales. Automatic control is ensured by a comprehensive instrumentation system.

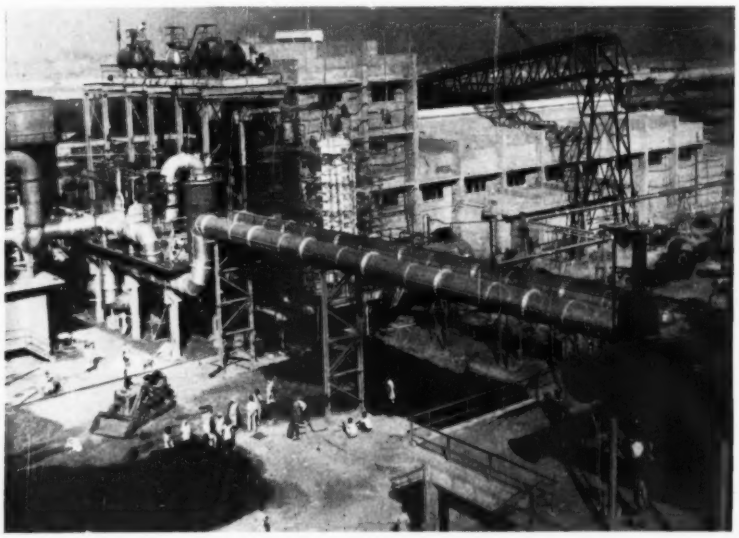
UNITED STATES

N Fertilizer with Zinc

A new nitrogen and zinc fertilizer has been marketed by Collier Carbon and Chemical Corp., Los Angeles, U.S.A. According to Collier, the formulation of the product Brea Brand Azua-Z has previously been unavailable to the agricultural industry.

Extensive field testing has convinced Collier that zinc is the most widespread micronutrient deficiency in Western States, and that next to nitrogen it is more generally deficient than any other element. It has been noticeable for some time that small but increasing quantities of certain metals—copper, iron, manganese, molybdenum and zinc—are used in some countries and areas where the soils are deficient in some of the plant micronutrient elements such as those named above, salts (namely the sulphate) of these elements being added to the fertilizers has resulted in significantly increased crop yields. In parts of the U S.A., and particularly the Western States, salts of boron, copper, zinc and manganese have been added to mixed fertilizers.

Deficiencies of zinc, iron and manganese occur most often on alkaline or overlimed soils, although exceptions occur, as in Southern Australia where zinc deficiencies may also occur on acid or neutral soils. Another factor in zinc deficiency is that the element is held



Ammonium sulphate plant at Durgapur which utilises by-product ammonia from coking operations

strongly by soils, so that recovery in the first crop is seldom over 5%. Use of nitrogen, particularly continued use of acid-forming ammonium sulphate without liming, can make soils which were formerly somewhat alkaline, extremely acid. The new Collier fertilizer by combining nitrogen in a suitable form could provide the necessary acidity for zinc to be made available as a plant nutrient as well as overcoming at the same time any nitrogen deficiency.

New Route to Concentrated N-P Fertilizer

A new N-P concentrated fertilizer from elemental phosphorus, air and ammonium has been produced in the U.S.A. on a laboratory scale. The substance is of particular interest since it contains N-P bonds. As early as 1890, it was suggested that phosphonitrilic compounds might be good sources of nitrogen and phosphorus for plants.

To produce the new compound, phosphorus vapour is burned in air and the phosphoric acid so obtained is ammoniated. The reaction is completed within the temperature range 250° to 350°C, the optimum temperature being about 315°C. The final product is a white fluffy material containing 17% N and 80% P_2O_5. It is believed to be a mixture of ammonium metaphosphate, phosphonitrilic acid and its ammonium salt. It has a low hygroscopicity, and on moistening and exposing to atmosphere is reported not to cake. The material is completely water-soluble, however.

Greenhouse experiments have shown that this new fertilizer material gives responses similar to those obtained with equal quantities of superphosphate and ammonium nitrate. As indicated by its effects on succeeding crops, it is notable that its residual value is high.

In evaluating this new concentrated fertilizer, the chemical engineering problems which are likely to be encountered in scaling-up the process from the laboratory scale to full-scale production plant must be considered. These could well be formidable ones.

CENTRAL AMERICA

El Salvador Fertilizer Site Chosen

Finalisation of plans for the fertilizer project initiated several years ago for El Salvador seems near at hand. Under the name Fertica (Fertilizantes de Centro America S.A.) with a group of Central American investors holding 50% of the capital and with U.S. and German interests participating in the balance, a company has been formed and is planning to build a U.S. $10 million fertilizer complex at Acajutla, El Salvador. The U.S. and German investors are International Development and Investment Co. Ltd. (I.D.I.) formed by Phoenix-Rheinrohr A.G., Dusseldorf, Germany (from the Thyssen-Hutte group, the largest steel makers in Europe), and the Chemical and Industrial International Ltd. of Nassau, Bahamas. The remaining 50% of equity capital required is from investors from El Salvador, Costa Rica and Panama. President of Fertica is a leading Salvadorean businessman, Mr. Francisco de Sola, and the general manager, Mr. Forge Marinel Dengo, a Costa Rican. It is intended that investors from the other Central American countries will be invited to participate

The plant will be designed to produce 180,000 tons of various types of high concentration fertilizers, enough to satisfy the demand of all the Central American Republics and Panama. Main installations will include a plant for production of high concentration nitrogen, phosphorus and potassium fertilizers with a capacity of 400 tons a day, a plant to produce 250 tons of nitric acid daily and an ammonium nitrate plant with a capacity of 150 tons daily.

While there are adequate supplies of hydroelectricity in El Salvador at present, there are no raw materials (natural gas, petroleum derivatives, coal, etc.) for production of chemical fertilizers. Available sources of supply at better prices than those from Europe and U.S.A. are available, however, in neighbouring Latin American countries. In due course an ammonia plant will be erected at Acajutla, but during the first states the fertilizer plant will use imported ammonia. The ammonia will come, it is now reported, from Cartagena, Colombia, from the ammonia plant being built by International Petroleum (Colombia) Ltd. (Intercol), a subsidiary of Standard Oil, New Jersey, U.S.A.

Present fertilizer imports by El Salvador total 69,000 tons a year valued at U.S. $5\frac{1}{2}$ million. It is intended that output of the new fertilizer works should meet virtually all the domestic requirements and be able to export to neighbouring Central American countries.

Statistical Appendix

West Germany Exports

Ammonium Sulphate Nitrate and Calcium Cyanamide

Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Saarland	480	—
Belgium	24,713	49,129
Denmark	13,026	4,085
Finland	1,701	583
France	3,000	8,969
Greece	18,877	15,234
Great Britain	31,453	38,656
Irish Republic	17,361	6,199
Luxemburg	617	215
Netherlands	2,140	272
Austria	1,871	544
Portugal	8,098	4,123
Sweden	5,418	390
Switzerland	336	486
Spain	173,510	49,483
Turkey	24,985	2,098
Egypt	934	77
Angola	693	1,431
Guinea Sp.	—	99
Cameroon Fr.	—	452
Canary Islands	32,727	12,797
Kenya & Uganda	1,121	2,099
Belgian Congo	519	85
Libya	892	297
Morocco	754	763
Nigeria	470	81
Portuguese E. Africa	17	1,581
Rhodesia & Nyasaland	13,158	7,598
Sudan	6,354	1,697
Union of South Africa	27,260	12,213
Tanganyika	1,239	88
Canada	293	300
Netherlands Antilles	—	230
Costa Rica	1,096	920
Dominican Republic	181	172
British Honduras	—	226
Cuba	2,359	1,814
Mexico	9,683	5
Nicaragua	10	247
Puerto Rico	1,814	36,907
El Salvador	20,673	12,212
West Indies	7,390	1,446
Argentina	5,677	2,681
Brazil	51,250	19,590
Chile	123	120
British Guiana	—	8,993
Colombo	2,143	296
Peru	15,484	22,823
Surinam	154	128
Uruguay	35	252
Venezuela	8,129	4,639
Burma	1,795	—
Ceylon	101	102
China	223,750	50,858
Cyprus	—	355
Hong Kong	—	149
India	3	99
Indonesia	10,373	46,296
Iraq	664	148
Iran	248	964
Jordan	1,440	585
South Korea	50,104	—
Lebanon	3,815	1,730
Malaya	7,254	2,056
Philippines	8,508	907
Singapore	1,628	648
Syria	1,315	853
Thailand	2,375	1,180
South Vietnam	5,774	422
New Zealand	1,650	772
Pakistan	201	—
Nepal	99	—
Iceland	118	—
Total	**862,249**	**440,851**

Other Nitrogenous Fertilizers

Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Portugal	712	—
Liberia	59	178
Morocco	250	—
Zanzibar	1,315	2,076
Union of South Africa	1,051	464
U.S.A.	2,640	429
Costa Rica	264	225
Mexico	2,508	2,497
Nicaragua	490	134
West Indies	—	59
Brazil	4,061	1,493
Ecuador	1,276	594
Colombia	3,846	2,799
Peru	5,223	—
Uruguay	—	125
Hong Kong	93	135
Malaya	1,154	496
Philippines	4,707	1,677
Singapore	813	311
Thailand	400	240
Australia	370	170
Portuguese E. Africa	98	—
Honduras	229	—
Total	**31,701**	**13,436**

Sodium Nitrate

Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Portugal	541	495
Rhodesia & Nyasaland	1,565	998
Greece	7,004	8,771
Union of South Africa	91	136
Brazil	1,564	301
Australia	1,495	—
New Zealand	1,315	305
China	202	—
Syria	353	196
U.S.A.	1,000	2,080
United Kingdom	—	297
Zanzibar	—	629
Mexico	—	298
Taiwan	—	200
Jordan	—	98
Others	1,225	—
Total	**16,355**	**13,634**

West Germany Exports

Ammonium Sulphate Nitrate and Calcium Nitrate

Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Bulgaria	44,250	72,564
Denmark	10,487	3,705
Finland	42,389	16,988
Greece	28,924	35,638
Great Britain	—	223
Irish Republic	4,503	559
Iceland	6,047	—
Yugoslavia	66,778	22,352
Luxemburg	976	433
Netherlands	23,074	9,318
Portugal	18,241	547
Spain	63,389	86,235
Turkey	4,359	737
Uruguay	—	25,299
Egypt	—	23,849
Ethiopia	807	603
Canary Islands	7,820	9,932
Kenya & Uganda	2,527	2,549
Libya	—	134
Morocco	—	315
Rhodesia & Nyasaland	14,153	11,264
Zanzibar	582	394
Sudan	—	744
Union of South Africa	7,775	10,792
Tanganyika	—	207
U.S.A.	16,023	7,275
Costa Rica	2,418	177
Guatemala	83	457
Mexico	30,383	25
Nicaragua	—	131
Panama	—	91
Puerto Rico	2,000	—
El Salvador	540	434
Br. West Indies	447	520
Argentina	1,276	615
Brazil	16,361	9,481
Ecuador	613	686
Colombia	835	498
Venezuela	136	87
Ceylon	—	123
China	71,775	8
Cyprus	206	372
India	76,589	49,288
Iran	138	148
Jordan	66	127
South Korea	503	—
Lebanon	3,465	1,749
Malaya	—	89
Philippines	—	433
Singapore	—	168
Syria	6,801	1,735
Thailand	100	215
Australia	5,311	996
New Zealand	1,012	358
Iraq	188	—
Cuba	907	—
Saarland	160	—
Belgium	305	—
Total	**799,665**	**549,570**

Ammonium Nitrate

Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Belgium	108	255
Finland	55	102
Greece	790	145
Luxemburg	41	132
Portugal	303	301
Morocco	2,051	400
Tunisia	100	100
Peru	360	281
India	—	9
Thailand	—	121
Others	102	99
Total	**3,910**	**2,044**

Chile

Exports of Nitrate

Country	1958	Jan. to Sept. 1959
Australia	13,135	3,709
Belgium	27,384	5,700
Canada		363
Denmark	16,000	8,050
Egypt	38,035	50,251
France	101,400	40,900
Germany	29,860	5,300
Greece	9,965	
India	16,271	19,136
Ireland	3,628	6,177
Italy	22,090	11,950
Japan	10,847	6,724
Jordan	297	297
Lebanon	2,174	4,640
Mexico	8,300	4,399
Netherlands	29,196	21,918
New Zealand	2,954	1,815
Portugal	23,624	11,501
Spain	198,494	111,097
Sweden	32,262	13,878
Syria	494	1,185
United Kingdom	13,037	4,878
United States	438,087	382,540
Yugoslavia	9,884	
London-in-transit items	6,020	41,324
Central America :		
Cuba	7,116	4,876
Costa Rica	997	
El Salvador	2,474	989
Guatemala	296	397
Jamaica	4	
Nicaragua	650	250
Panama	20	
South America :		
Argentina	31,221	20,495
Bolivia	32	
Brazil	54,104	41,270
Colombia	2,135	161
Ecuador	1,252	1,048
Paraguay		5
Peru	16,875	7,271
Uruguay	3,094	278
Venezuela	660	879
Other countries		20,260
Total	**1,175,372**	**1,855,911**

Statistical Appendix

West Germany Exports

Ammonium Sulphate Nitrate and Calcium Cyanamide

Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
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Cuba	2,359	1,814
Mexico	9,683	5

Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Nicaragua	10	247
Puerto Rico	1,814	36,907
El Salvador	20,673	12,212
West Indies	7,390	1,446
Argentina	5,677	2,681
Brazil	51,250	19,590
Chile	123	120
British Guiana	—	8,993
Colombo	2,143	296
Peru	15,484	22,823
Surinam	154	128
Uruguay	35	252
Venezuela	8,129	4,639
Burma	1,795	—
Ceylon	101	102
China	223,750	50,858
Cyprus	—	355
Hong Kong	—	149
India	3	99
Indonesia	10,373	46,296
Iraq	664	148
Iran	248	964
Jordan	1,440	585
South Korea	50,104	—
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Singapore	1,628	648
Syria	1,315	853
Thailand	2,375	1,180
South Vietnam	5,774	422
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Pakistan	201	—
Nepal	99	—
Iceland	118	—
Total	**862,249**	**440,851**

Other Nitrogenous Fertilizers

Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Portugal	712	—
Liberia	59	178
Morocco	250	—
Zanzibar	1,315	2,076
Union of South Africa	1,051	464
U.S.A.	2,640	429
Costa Rica	264	225
Mexico	2,508	2,497
Nicaragua	490	134
West Indies	—	59
Brazil	4,061	1,493
Ecuador	1,276	594
Colombia	3,846	2,799
Peru	5,223	—
Uruguay	—	125
Hong Kong	93	135
Malaya	1,154	496
Philippines	4,707	1,677
Singapore	813	311
Thailand	400	240
Australia	370	170
Portuguese E. Africa	98	—
Honduras	229	—
Total	**31,701**	**13,436**

Sodium Nitrate

Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Portugal	541	495
Rhodesia & Nyasaland	1,565	998
Greece	7,004	8,771
Union of South Africa	91	136
Brazil	1,564	301
Australia	1,495	—
New Zealand	1,315	305
China	202	—
Syria	353	196
U.S.A.	1,000	2,080
United Kingdom	—	297
Zanzibar	—	629
Mexico	—	298
Taiwan	—	200
Jordan	—	98
Others	1,225	—
Total	**16,355**	**13,634**

West Germany Exports

Ammonium Sulphate Nitrate and Calcium Nitrate

Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960	Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Bulgaria	44,250	72,564	Panama	—	91
Denmark	10,487	3,705	Puerto Rico	2,000	—
Finland	42,389	16,988	El Salvador	540	434
Greece	28,924	35,638	Br. West Indies	447	520
Great Britain	—	223	Argentina	1,276	615
Irish Republic	4,503	559	Brazil	16,361	9,481
Iceland	6,047	—	Ecuador	613	686
Yugoslavia	66,778	22,352	Colombia	835	498
Luxemburg	976	433	Venezuela	136	87
Netherlands	23,074	9,318	Ceylon	—	123
Portugal	18,241	547	China	71,775	8
Spain	63,389	86,235	Cyprus	206	372
Turkey	4,359	737	India	76,589	49,288
Uruguay	—	25,299	Iran	138	148
Egypt	—	23,849	Jordan	66	127
Ethiopia	807	603	South Korea	503	—
Canary Islands	7,820	9,932	Lebanon	3,465	1,749
Kenya & Uganda	2,527	2,549	Malaya	—	89
Libya	—	134	Philippines	—	433
Morocco	—	315	Singapore	—	168
Rhodesia & Nyasaland	14,153	11,264	Syria	6,801	1,735
Zanzibar	582	394	Thailand	100	215
Sudan	—	744	Australia	5,311	996
Union of South Africa	7,775	10,792	New Zealand	1,012	358
Tanganyika	—	207	Iraq	188	—
U.S.A.	16,023	7,275	Cuba	907	—
Costa Rica	2,418	177	Saarland	160	—
Guatemala	83	457	Belgium	305	—
Mexico	30,383	25	**Total**	**799,665**	**549,570**
Nicaragua	—	131			

Ammonium Nitrate

Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960	Country	First Half Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Belgium	108	255	Peru	360	281
Finland	55	102	India	—	9
Greece	790	145	Thailand	—	121
Luxemburg	41	132	Others	102	99
Portugal	303	301			
Morocco	2,051	400	**Total**	**3,910**	**2,044**
Tunisia	100	100			

Chile

Exports of Nitrate

Country	1958	Jan. to Sept. 1959	Country	1958	Jan. to Sept. 1959
Australia	13,135	3,709	London-in-transit items	6,020	41,324
Belgium	27,384	5,700	Central America :		
Canada		363	Cuba	7,116	4,876
Denmark	16,000	8,050	Costa Rica	997	
Egypt	38,035	50,251	El Salvador	2,474	989
France	101,400	40,900	Guatemala	296	397
Germany	29,860	5,300	Jamaica	4	
Greece	9,965		Nicaragua	650	250
India	16,271	19,136	Panama	20	
Ireland	3,628	6,177	South America :		
Italy	22,090	11,950	Argentina	31,221	20,495
Japan	10,847	6,724	Bolivia	32	
Jordan	297	297	Brazil	54,104	41,270
Lebanon	2,174	4,640	Colombia	2,135	161
Mexico	8,300	4,399	Ecuador	1,252	1,048
Netherlands	29,196	21,918	Paraguay		5
New Zealand	2,954	1,815	Peru	16,875	7,271
Portugal	23,624	11,501	Uruguay	3,094	278
Spain	198,494	111,097	Venezuela	660	879
Sweden	32,262	13,878	Other countries		20,260
Syria	494	1,185			
United Kingdom	13,037	4,878	**Total**	**1,175,372**	**1,855,911**
United States	438,087	382,540			
Yugoslavia	9,884				

Netherlands Exports

Ammonium Sulphate

Country	Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Sweden	3,030	1,118
Portugal	—	990
Spain	110,775	10,800
Greece	9,999	10,370
Libya	198	990
Canary Islands	9,900	3,465
Union of South Africa	2,826	5,326
Rhodesia	612	—
Salvador	1,458	2,419
Cyprus	—	574
Venezuela	237	197
Surinam	1,733	115
Uruguay	—	980
Argentina	3,324	2,759
Indonesia	16,387	—
Ceylon	26,064	—
China	40,366	15,515
New Zealand	1,394	845
Ireland	24,287	6,419
Nigeria	98	—
Portuguese E. Africa	20	—
Philippines	—	34,267
Guatemala	139	102
Jamaica	4,427	4,862
Brazil	4,765	1,363
Thailand	2,850	4,800
Malaya	—	522
Vietnam	1,966	—
Belgium	83	—
India	—	19,092
West Germany	400	—
Leeward Islands	—	279
Belgium/Luxemburg	84	15
Great Britain	6,320	1,523
Turkey	4,391	—
Peru	1,500	500
Cuba	—	5,080
Hungary	—	20,610
Others	911	—
Total	**280,544**	**154,293**

Ammonium Phosphate

Country	Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Belgium/Luxembourg	120	20
Great Britain	437	1,495
Ireland	—	426
Libya	327	—
Nigeria	27	—
Union of South Africa	45	27
Trinidad	50	25
Malaya	30	35
West Germany	—	30
Sweden	—	57
Mauritius	90	45
Indonesia	202	1
Ceylon	—	35
Thailand	120	—
Uruguay	92	140
Finland	—	49
Liberia	—	70
Chile	—	20
Others	115	—
Total	**1,735**	**2,316**

Urea

Country	Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Belgium/Luxembourg	400	654
United Kingdom	41	32
France	—	161
Ireland	41	—
Sweden	1,463	2,729
Denmark	52	85
Portugal	779	55
Spain	625	425
Greece	65	70
Poland	151	—
Sudan	5,273	—
Belgian Congo	374	129
Uganda	295	26
Kenya	148	—
Tanganyika	122	240
Portuguese E. Africa	454	—
Rhodesia	574	1,735
U.K. Africa	2,575	5,657
U.S.A.	8,485	795
Guatemala	233	212
British Honduras	59	3,159
Salvador	248	303
Nicaragua	—	99
Costa Rica	362	167
Panama	59	154
Colombia	116	656
British Guiana	1,005	508
Ecuador	197	—
Brazil	2,823	709
Argentina	1,203	229
India	—	1,036
Ceylon	158	30
Malaya	30	—
Philippines	641	7,597
China	11,329	—
Australia	20,349	—
Finland	—	196
Czechoslovakia	—	50
Canary Islands	—	133
Mexico	1,270	33
Honduras	1,419	8,612
Dominican Republic	—	44
Porto Rica	—	91
Venezuala	—	124
Peru	500	—
Uruguay	—	103
Iran	—	850
South Korea	—	27
Others	499	454
Total	**63,004**	**39,297**

Calcium Nitrate

Country	Fertilizer Year 1958/1959	First Half Fertilizer Year 1959/1960
Belgium/Luxembourg	4,123	1,543
Sweden	32,364	7,460
Yugoslavia	19,342	—
U.S.A.	7,040	5,920
Indonesia	206	—
Portugal	2,371	397
Lebanon	—	99
Total	**65,451**	**15,318**

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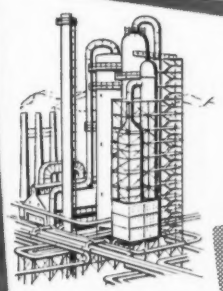

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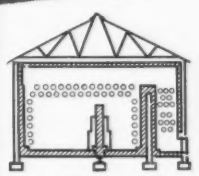

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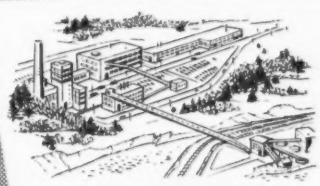

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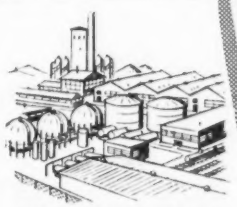

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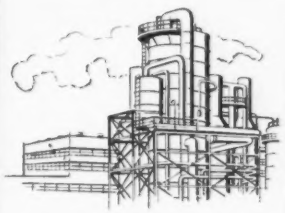

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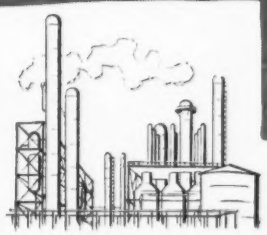

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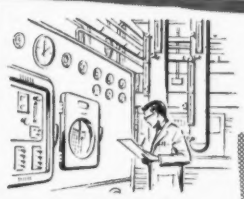

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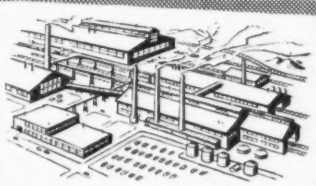

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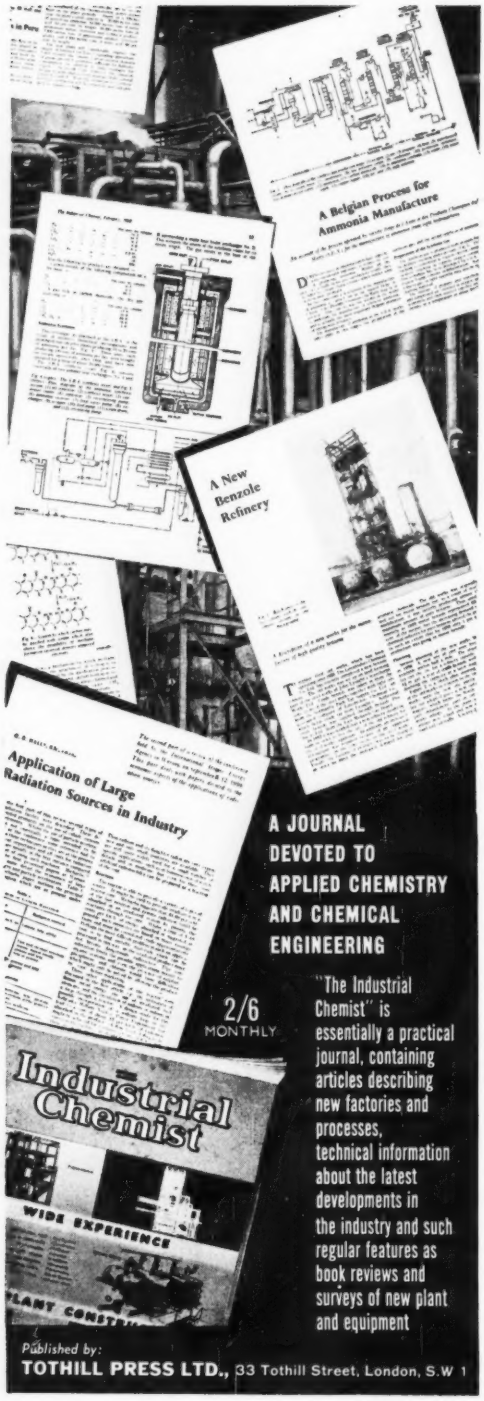
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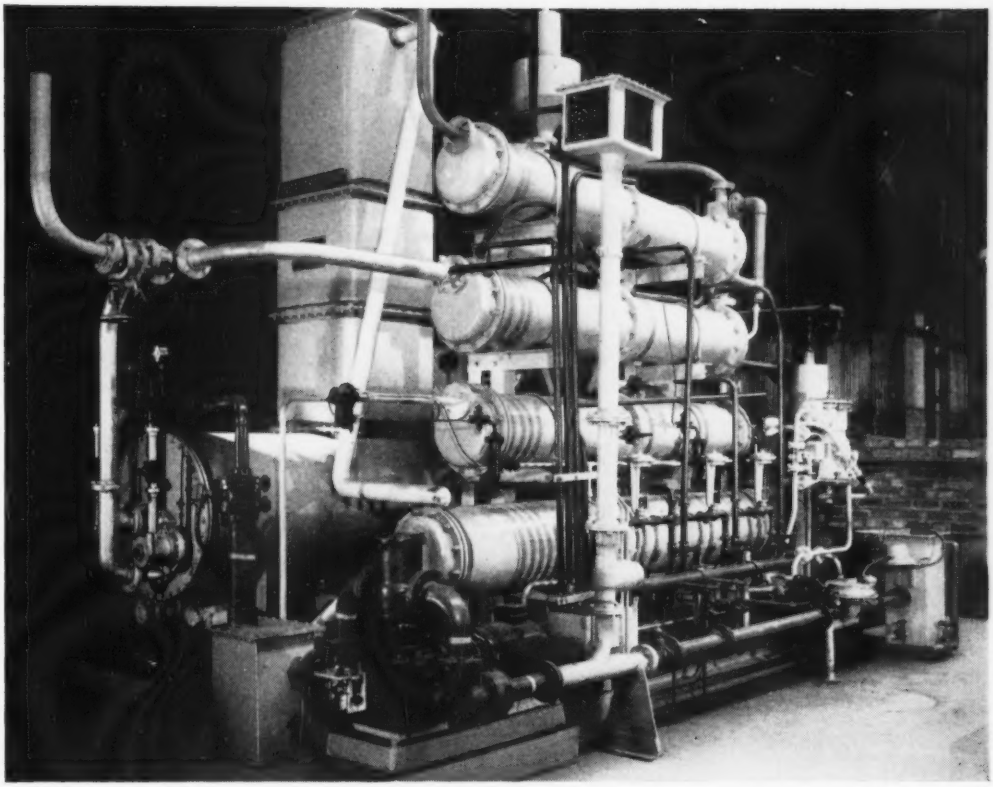

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